Analytical Abstracts

A monthly publication dealing with
all branches of analytical chemistry

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ANALYTICAL SCIENCES MONOGRAPHS No. 3

Pyrolysis–Gas Chromatography

by R. W. May, E. F. Pearson and D. Scothern

This monograph attempts to present the available knowledge in a form useful to the practising analyst, helping in the choice of an appropriate method and in the avoidance of the more common pitfalls in this, perhaps deceptively, simple technique.

Chapter 1 serves as an introduction to gas chromatography and will be of interest to those unfamiliar with the technique. The several methods of pyrolysis used in pyrolysis–gas chromatography are described in Chapter 2; their merits and demerits in particular applications are discussed. The major analytical uses of the technique are presented in Chapter 3; the general analytical 'fingerprinting' aspects described separately from the method as applied to specific sample types. Chapter 4 deals with the identification of the pyrolysis products which are eluted from the chromatography column, useful extra information allowing the possibility of naming a pyrolysed sample without recourse to a known identical sample. The necessity for increased standardization of the technique of pyrolysis–gas chromatography is discussed in Chapter 5.

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CS Members £5.50

ANALYTICAL SCIENCES MONOGRAPHS No. 4

Electrothermal Atomization for Atomic Absorption Spectrometry

by C. W. Fuller

At the present time the two most successful alternatives to the flame appear to be the electrothermal atomizer and the inductively-coupled plasma. In this book an attempt has been made to provide the author's views on the historical development, commercial design features, theory, practical considerations, analytical parameters of the elements, and areas of application of the first of these two techniques, electrothermal atomization.

The chapter headings are as follows: History; Theoretical Aspects of the Atomization Process; General Experimental Conditions; Analytical Conditions for the Determination of the Elements by Atomic Absorption Spectrometry; Applications (Oil and Oil Products; Metals; Rocks, Minerals, and Soils; Waters; Plants; Food and Drugs; Biological Fluids; Biological Tissues; Air Particulates; Refractory Oxides and Related Materials; Other Analytical Applications; Theoretical).

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THE CHEMICAL SOCIETY

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Introduction to Analytical Abstracts

Scope and Selection

Analytical Abstracts endeavours to cover the whole field of analytical chemistry. In more than 8500 abstracts a year it surveys the world's literature and selects for abstracting those papers and other publications that are considered to be of interest and importance to the analytical chemist. Certain sources, in particular dissertations and patents, are excluded.

Sections

The abstracts are grouped into the 9 main sections shown below. Within each section or sub-section an inner ordering is followed so that abstracts on similar subjects should be found together. An example of this ordering is the Periodic-Group order of the elements in Section B—Inorganic Chemistry. In general, abstracts on specific analyses are classified according to the sample matrix. At the end of each main section, after book titles, cross-references to relevant abstracts in other sections are given.

Section headings

A—General analytical chemistry
B—Inorganic chemistry
C—Organic chemistry
D—Biochemistry
E—Pharmaceutical chemistry
F—Food
G—Agriculture
H—Air; water; effluents
J—Techniques; apparatus

Abstracts

The following abstract contains most of the features:

① ② 2A13. **Standardisation of quantitative analytical procedures. V. Gravimetric determinations.** ③ Gottschalk, G. (④ Osram G.m.b.H., Muenchen, W. Germany). ⑤ *Fresenius Z. Anal. Chem.*, 1976, **280** (3), 205–218 (in German). The principles of the standardisation method previously described (*Ibid.*, 1975, **275**, 1; 1975, **276**, 81, 257; 1976, **278**, 1) were applied to the ⑥ gravimetric determination of W (0·1 to 1 mmol) by pptn. with 8-hydroxyquinoline. The optimum composition of the test soln. and the standard soln., the standardisation procedure, 'blank' values, control tests, computer evaluation and interpretation of results, and selectivity are discussed. The information obtained was used in establishing a procedure for the determination of W in scheelite. (For Part IV see *Anal. Abstr.*, 1976, **31**, 2A10.) ⑦ W.C.D.

① ***Abstract number***—The abstract designation consists of three parts (left to right):

(a) *Issue Number*. A number 1 to 6 corresponding to the monthly issue number in the volume. (There are two volumes of six separate monthly issues each year.)

(b) *Section Letter*. Each subject section is indicated by a letter of the alphabet A to J (the letter I is avoided to prevent possible confusion).

(c) *Abstract Number*. An individual number of the abstract within its section. The numbers start with 1 in each section of each issue and run consecutively throughout the section.

② ***Title***—The title is given in full if in English in the original, or in translation if in a foreign language.

③ ***Authors***—Names of all authors are given as in the original or, if in Cyrillic script, transliterated according to BS 2979:1958 (British System). The German ä, ö and ü and the Danish and Norwegian ø are rendered as ae, oe, ue and oe respectively. The address given in parentheses is, in general, that at which the work was carried out.

④ ***Source***—Titles of journals are abbreviated as in the *Chemical Abstracts Service Source Index* in accordance with the standards of the International Organization for

Standardization (ISO). The language of the original paper, if other than English, is indicated in parentheses. A list of the main journals scanned is given on page vi of this introduction.

⑤ ***Abstract***—In general, no attempt is made to give a full working abstract of the method of analysis. The amount of detail, however, is largely determined by the accessibility and language of the source material.

⑥ ***References***—References to previous publications or to abstracts in Analytical Abstracts are given where these provide additional relevant information.

⑦ ***Credits***—Abstractors are identified by initials; a list of names is given below.

Abstractors

A.B.K.S.	A. B. Knight-Sweeney	J.W.P.	J. W. Price
A.B.S.	A. Burwood-Smith	J.Z.	J. Zyka
A.McC.	A. McCraight	K.S.	K. Saito
A.R.R.	A. R. Rogers	L.A.O'N.	L. A. O'Neill
A.S.W.	A. S. Wilson	L.B.	L. Barton
B.D.H.	B. D. Hemmings	L.D.A.	L. D. Arnold
B.H.	B. Harris	L.M.C.	L. M. Cecil
B.K.	B. Kaminski	L.S.R.	L. S. Rao
C.A.H.	C. A. Helsby	M.A.P.	M. A. Pringuer
C.B.	C. Brown	M.B.G.	M. B. Gill
C.D.K.	C. D. Kopkin	M.J.Sa.	M. J. Saxby
C.K.L.	C. K. Laird	M.J.Sl.	M. J. Slucutt
D.B.A.	D. B. Adams	M.P.	M. Polasek
D.I.R.	D. I. Rees	M.R.M.	M. R. Maner
D.W.S.	D. W. Selby	M.W.G.B.	M. W. G. Burt
E.A.	E. Aspray	O.B.	O. Ballin
E.C.A.	E. C. Apling	P.B.	P. Brych
E.G.B.	E. G. Brickell	P.C.R.	P. C. Rouse
E.G.N.M.	E. G. N. Marsden	P.M.B.	P. M. Brown
E.J.H.B.	E. J. H. Birch	R.A.H.	R. A. Hendey
E.K.O.	E. K. Orry	R.F.S.	R. F. Smith
F.P.	F. Powell	R.J.F.	R. J. Ferguson
F.R.B.	F. R. Basford	R.L.J.	R. L. Jordan
G.B.	G. Burger	R.P.E.	R. P. Earthy
G.E.F.	G. E. Foster	R.W.	R. Waspe
G.J.H.	G. J. Hunter	R.W.L.	R. W. Lines
G.P.C.	G. P. Cook	S.C.	S. Castles
G.R.	G. Russell	S.E.P.	S. E. Pal
G.S.S.	G. S. Smith	S.K.	S. Kaliski
H.I.	H. M. N. H. Irving	S.M.M.	S. M. Marsh
H.K.M.	H. K. Morgan	T.G.A.	T. G. Alliston
H.L.L.	H. L. Lehmann	T.H.N.	T. H. Newman
H.M.T.	H. M. Thompson	T.J.S.	T. J. Samuelson
I.R.H.C.	I. R. H. Crail	V.B.	V. Bernstein
J.C.	J. Cryer	V.S.	V. Smith
J.E.P.	J. E. Page	W.C.D.	W. Cule Davies
J.H.A.R.	J. H. A. Ruzicka	W.C.J.	W. C. Johnson
J.K.C.	J. K. Corbett	W.H.	W. Houston
J.M.B.	J. M. Brown	W.J.B.	W. J. Baker
J.P.A.	J. P. Arnold		

Abbreviations and Symbols

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

adenosine 5′-monophosphate	AMP
adenosine 5′-pyrophosphate	ADP
adenosine 5′-triphosphate	ATP
alternating current	a.c.
approximate, -ly	approx.
aqueous	aq.
atomic-absorption spectrophotometr-y, -ic	a.a.s.
atomic-emission spectrometr-y, -ic	a.e.s.
atomic-fluorescence spectrometr-y, -ic	a.f.s.
atomic number	Z
attenuated total reflectance	a.t.r.
biochemical oxygen demand	BOD
boiling-point	b.p.
chemical oxygen demand	COD
coefficient	coeff.
concentrated	conc.
concentration	concn.
crystalline / crystallised	cryst.
current density	c.d.
density, relative	d
deoxyribonucleic acid	DNA
diameter, inside	i.d.
diameter, outside	o.d.
differential thermal analysis	d.t.a.
dilute (as adjective)	dil.
5-dimethylaminonaphthalene-1-sulphonyl	dansyl
direct current	d.c.
dissociation constant	K
electron spin (or paramagnetic) resonance	e.s.r.
ethylenediaminetetra-acetate	edta
gas chromatograph-y, -ic	g.c.
gas - liquid chromatograph-y, -ic	g.l.c.
gas - solid chromatograph-y, -ic	g.s.c.
half-wave potential	$E_{\frac{1}{2}}$
height equivalent to a theoretical plate	h.e.t.p.
high-performance liquid chromatography	h.p.l.c.
infra-red	i.r.
insoluble	insol.
ionic strength	I
mass spectrometr-y, -ic	m.s.
maxim-um, -a	max.
molar absorption coefficient	ϵ
molecular weight	mol. wt.
neutron-activation analysis	n.a.a.
nicotinamide–adenine dinucleotide (oxidised form)	NAD^+
nicotinamide–adenine dinucleotide (reduced form)	NADH
normal calomel electrode	n.c.e.
normal hydrogen electrode	n.h.e.
nuclear magnetic resonance	n.m.r.
optical rotation	α
optical rotatory dispersion	o.r.d.
partial pressure (as in, *e.g.*, $p CO_2$)	p
poly(tetrafluoroethylene)	PTFE
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitation	pptn.
preparation	prep.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
ribonucleic acid	RNA
saturated (as adjective)	satd.
saturated calomel electrode	s.c.e.
soluble	sol.
solution	soln.
specific rotation	$[\alpha]_\lambda^t$
standard temp. and pressure	s.t.p.
thin-layer chromatograph-y, -ic	t.l.c.
ultra-violet	u.v.
volume	vol.
wavelength	λ
weight	wt.

Units of Measurement

As well as those listed below, other symbols given in the 'Système International d'Unités' (SI) may be used where appropriate. The symbols 'mμ' (for nm) and 'γ' (for μg) are not used.

ampere	A
ångström	Å
atmosphere	atm
atomic mass unit	a.m.u.
counts per minute	c.p.m.
counts per second	c.p.s.
curie	Ci
disintegrations per minute	d.p.m.
electron-volt	eV
equivalent	equiv
farad	F
gauss	G
gram	g
gram-molecule	mol
hertz	Hz
hour	h
international unit	i.u.
joule	J
kelvin*	K
kilogram	kg
litre	l
metre	m
minute	min
molar (concentration)	M

* Temperatures given in degrees are on the Celsius scale unless otherwise indicated.

neutrons per square centimetre per second	n cm^{-2} s^{-1}	pounds per square inch	p.s.i.
newton	N	revolutions per minute	r.p.m.
normal (concentration)	N	roentgen	R
ohm	Ω	second (time)	s
parts per U.S. billion (10^9)	p.p.b.	siemens	S
parts per million	p.p.m.	tesla	T
pascal	Pa	tonne (10^6 g)	t
per cent. (vol. in vol.)	%(v/v)	unit of pressure (mm Hg)	Torr
per cent. (wt. in vol.)	%(w/v)	volt	V
per cent. (wt. in wt.)	%(w/w)	watt	W
		weber	Wb

The following prefixes may be used in conjunction with the symbols—

Fraction	*Prefix*	*Symbol*	*Multiple*	*Prefix*	*Symbol*
10^{-1}	deci	d	10	deka	da
10^{-2}	centi	c	10^2	hecto	h
10^{-3}	milli	m	10^3	kilo	k
10^{-6}	micro	μ	10^6	mega	M
10^{-9}	nano	n	10^9	giga	G
10^{-12}	pico	p	10^{12}	tera	T
10^{-15}	femto	f			
10^{-18}	atto	a			

The chemical nomenclature used in the abstracts is that recommended by The International Union of Pure and Applied Chemistry ('Nomenclature of Organic Chemistry', sections A and B, 3rd Edn, and Section C, 2nd Edn, Butterworths, London, 1971; 'Nomenclature of Inorganic Chemistry', Butterworths, London, 1971).

When known, the Colour Index (C.I.) designations of dyes are generally included. The names given to enzymes are those recommended by The International Union of Pure and Applied Chemistry and the International Union of Biochemistry ('Enzyme Nomenclature', Elsevier Scientific Publishing Co., Amsterdam, 1973). When possible, drugs are specified by their official B.P. or B.P.C. names, or by the Approved Names recommended by the British Pharmacopoeia Commission. The principal Pharmacopoeias are denoted by, *e.g.*, B.P., U.S.P. or Ph.Eur., together with the identifying roman numeral or year. Pesticides are named in accordance with British Standard 1831:1969, 'Recommended Common Names for Pesticides', and addenda.

Indexes

A subject and author volume index covering 6 monthly issues is published twice a year as part of the annual subscription.

Cumulative indexes—Two cumulative indexes only are available. The first covers the 10 years 1954–1963 and the second covers the 5 years 1964–1968 inclusive. These may be purchased from The Chemical Society, Distribution Centre, Blackhorse Road, Letchworth, Herts., SG6 1HN.

Microfilm

Enquiries on the availability of microfilm versions of Analytical Abstracts should be addressed to:

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Serials Bid Coordinator
300 North Zeeb Road
Ann Arbor, Michigan 48106

Photocopies

Photocopies of most papers cited can be obtained from The Librarian, The Chemical Society, Burlington House, London W1V 0BN, to whom enquiries should be addressed. Prices are subject to revision but at the time of going to press are 8p. per page of original text (5p. per page to members of The Chemical Society).

Journals

The following list of journals includes all those that are scanned regularly. Abstracts of articles from primary sources not included in the list are sometimes reproduced by permission from the secondary sources indicated with an asterisk. Credit is given to the source with each individual abstract. Where papers from Russian journals are credited to *Referativnyi Zhurnal* the translated title and bibliographic descriptions only are given.

Abbreviations of journal titles used in the abstracts are based on ISO Standards as given in the *Chemical Abstracts Service Source Index*. The language(s) in which the journal is published is indicated in italics after the full name of the title.

**Abstr. Bulg. Sci. Lit., Chem.*	Abstracts of Bulgarian Scientific Literature, Chemistry [*Eng.*]
**Abstr. Bull. Inst. Pap. Chem.*	Abstract Bulletin of the Institute of Paper Chemistry [*Eng.*]
**Abstr. Flour Milling Baking Res. Assoc.*	Abstracts of the Flour Milling and Baking Research Association [*Eng.*]
**Abstr. Rom. Sci. Tech. Lit.*	Abstracts of Romanian Scientific and Technical Literature [*Eng.*]
Acta Chem. Scand.	Acta Chemica Scandinavica [*Eng., Fr., Ger.*]
Ser. A	Series A: Physical and Inorganic Chemistry
Ser. B	Series B: Organic Chemistry and Biochemistry
Acta Chim. Acad. Sci. Hung.	Acta Chimica Academiae Scientiarum Hungaricae [*Eng., Fr., Ger., Russ.*]
Acta Pharm. Hung.	Acta Pharmaceutica Hungarica [*Hung.*]
Acta Pharm. Jugosl.	Acta Pharmaceutica Jugoslavica [*Serbo-Croat*]
Acta Pharm. Suec.	Acta Pharmaceutica Suecica [*Eng.*]
Acta Pol. Pharm.	Acta Poloniae Pharmaceutica [*Pol.*]
Actual. Chim.	L'Actualité Chimique [*Fr.*]
Agric. Biol. Chem.	Agricultural and Biological Chemistry [*Eng.*]
AMDEL Bull.	AMDEL Bulletin (Australian Mineral Development Laboratories) [*Eng.*]
Am. J. Hosp. Pharm.	American Journal of Hospital Pharmacy [*Eng.*]
Am. J. Pharm.	American Journal of Pharmacy and the Sciences Supporting Public Health [*Eng.*]
Am. Lab.	American Laboratory [*Eng.*]
Anal. Biochem.	Analytical Biochemistry [*Eng.*]
Anal. Chem.	Analytical Chemistry [*Eng.*]
Anal. Chim. Acta	Analytica Chimica Acta [*Eng., Fr., Ger.*]
Anal. Lett.	Analytical Letters [*Eng., Fr., Ger.*]
Analusis	Analusis [*Eng., Fr.*]
Analyst (London)	Analyst [*Eng.*]
An. Bromatol.	Anales de Bromatologia [*Span.*]
An. Fis.	Anales de Fisica [*Eng., Span.*]
Angew. Chem., Int. Ed. Engl.	Angewandte Chemie, International Edition in English [*Eng.*]
Angew. Makromol. Chem.	Angewandte Makromolekulare Chemie [*Eng., Fr., Ger.*]
Ann. Biol. Anim., Biochim., Biophys.	Annales de Biologie Animale, Biochimie, Biophysique [*Eng., Fr.*]
Ann. Chim. (Paris)	Annales de Chimie [*Fr.*]
Ann. Chim. (Rome)	Annali di Chimica [*Eng., Ital.*]
Ann. Clin. Biochem.	Annals of Clinical Biochemistry [*Eng.*]
Ann. Falsif. Expert. Chim.	Annales de Falsification et de l'Expertise Chimique [*Fr.*]
Ann. Ist. Super. Sanita	Annali dell'Istituto Superiore di Sanita [*Eng., Fr., Ital.*]
Ann. Occup. Hyg.	Annals of Occupational Hygiene [*Eng.*]
Ann. Pharm. Fr.	Annales Pharmaceutiques Françaises [*Fr.*]
An. Quim.	Anales de Quimica [*Eng., Span.*]
Antibiotiki (Moscow)	Antibiotiki. Moscow [*Russ.*]
Appl. Opt.	Applied Optics [*Eng., Fr., Ger., Russ.*]
Appl. Phys. Lett.	Applied Physics Letters [*Eng.*]
Appl. Spectrosc.	Applied Spectroscopy [*Eng.*]
**Arab Sci. Abstr.*	Arab Science Abstracts [*Eng.*]
Arch. Eisenhuettenwes.	Archiv für das Eisenhüttenwesen [*Ger.*]
Arch. Environ. Contam. Toxicol.	Archives of Environmental Contamination and Toxicology [*Eng.*]
Arch. Pharm. (Weinheim, Ger.)	Archiv der Pharmazie [*Ger.*]
Arm. Khim. Zh.	Armyanskii Khimicheskii Zhurnal [*Russ.*]
Arzneim.-Forsch.	Arzneimittel-Forschung [*Eng., Ger.*]
At. Absorpt. Newsl.	Atomic Absorption Newsletter [*Eng.*]
At. Energ.	Atomnaya Energiya [*Russ.*]
Atmos. Environ.	Atmospheric Environment [*Eng.*]
Aust. J. Agric. Res.	Australian Journal of Agricultural Research [*Eng.*]
Aust. J. Chem.	Australian Journal of Chemistry [*Eng.*]
Azerb. Khim. Zh.	Azerbaidzhanskii Khimicheskii Zhurnal [*Azerb., Russ.*]

BCRA Rev.	BCRA Review [*Eng.*]
Beitr. Tabakforsch.	Beiträge zur Tabakforschung [*Eng., Ger.*]
Biochem. J.	Biochemical Journal [*Eng.*]
Biochem. Med.	Biochemical Medicine [*Eng.*]
Biochim. Biophys. Acta	Biochimica et Biophysica Acta [*Eng., Fr., Ger.*]
Biochimie	Biochimie [*Eng., Fr.*]
Biokhimiya	Biokhimiya [*Russ.*]
Biomed. Mass Spectrom.	Biomedical Mass Spectrometry [*Eng.*]
Bios (Nancy)	Bios. Nancy [*Fr.*]
Boll. Soc. Ital. Biol. Sper.	Bollettino della Societa Italiana di Biologia Sperimentale [*Ital.*]
Brauwissenschaft	Brauwissenschaft [*Ger.*]
Br. Med. J.	British Medical Journal [*Eng.*]
Bull. Acad. Pol. Sci., Ser. Sci. Chim.	Bulletin de l'Académie Polonaise des Sciences, Série des Sciences Chimiques [*Eng., Fr., Ger.*]
Bull. Chem. Soc. Jpn.	Bulletin of the Chemical Society of Japan [*Eng., Ger.*]
Bull. Coll. Sci., Univ. Baghdad	Bulletin of the College of Science, University of Baghdad [*Eng.*]
Bull. Environ. Contam. Toxicol.	Bulletin of Environmental Contamination and Toxicology [*Eng.*]
Bull. Haffkine Inst.	Bulletin of Haffkine Institute [*Eng.*]
Bull. Inst. Chem. Res., Kyoto Univ.	Bulletin of the Institute for Chemical Research, Kyoto University [*Eng.*]
Bull. Narc.	Bulletin on Narcotics [*Eng., Span., Russ.*]
Bull. Soc. Chim. Belg.	Bulletin des Sociétés Chimiques Belges [*Fr.*]
Bull. Soc. Chim. Fr.	Bulletin de la Société Chimique de France [*Fr.*]
Bunko Kenkyu	Bunko Kenkyu (Journal of the Spectroscopical Society of Japan) [*Eng., Jap.*]
Bunseki Kagaku	Bunseki Kagaku (Japan Analyst) [*Jap.*]
**Cadmium Abstr.*	Cadmium Abstracts [*Eng.*]
Can. J. Biochem.	Canadian Journal of Biochemistry [*Eng., Fr.*]
Can. J. Chem.	Canadian Journal of Chemistry [*Eng., Fr.*]
Can. J. Pharm. Sci.	Canadian Journal of Pharmaceutical Sciences [*Eng., Fr.*]
Can. J. Physiol. Pharmacol.	Canadian Journal of Physiology and Pharmacology [*Eng., Fr.*]
Can. J. Spectrosc.	Canadian Journal of Spectroscopy [*Eng., Fr.*]
Cereal Chem.	Cereal Chemistry [*Eng.*]
Cereal Foods World	Cereal Foods World [*Eng.*]
Cesk. Farm.	Ceskoslovenska Farmacie [*Czech, Slovak*]
**Chem. Abstr.*	Chemical Abstracts [*Eng.*]
Chem. Age (London)	Chemical Age. London [*Eng.*]
Chem. Anal. (Warsaw)	Chemia Analityczna [*Eng., Ger., Pol., Russ.*]
Chem. Br.	Chemistry in Britain [*Eng.*]
Chem. Eng. News	Chemical & Engineering News [*Eng.*]
Chem. Geol.	Chemical Geology [*Eng., Fr., Ger.*]
Chem. Ind. (London)	Chemistry and Industry [*Eng.*]
Chem.-Ing.-Tech.	Chemie-Ingenieur-Technik [*Eng., Ger.*]
Chemist-Analyst	Chemist-Analyst [*Eng.*]
Chem. Lett.	Chemistry Letters [*Eng., Fr., Ger., Jap.*]
Chem. Listy	Chemicke Listy [*Czech*]
Chem. Pharm. Bull.	Chemical and Pharmaceutical Bulletin [*Eng., Fr., Ger.*]
Chem. Prum.	Chemicky Prumysl [*Czech*]
Chem. Scr.	Chemica Scripta [*Eng., Fr., Ger.*]
Chem. Soc. Rev.	Chemical Society Reviews [*Eng.*]
Chem.-Tech. (Heidelberg)	Chemie-Technik [*Ger.*]
Chem. Tech. (Leipzig)	Chemische Technik [*Ger.*]
Chem. Ztg.	Chemiker Zeitung [*Ger.*]
Chem. Zvesti	Chemicke Zvesti [*Eng., Fr., Ger., Russ.*]
Chimia	Chimia [*Eng., Fr., Ger.*]
Chromatographia	Chromatographia [*Eng., Fr., Ger.*]
Clin. Chem. (Winston-Salem, N.C.)	Clinical Chemistry [*Eng.*]
Clin. Chim. Acta	Clinica Chimica Acta [*Eng., Fr., Ger.*]
Collect. Czech. Chem. Commun.	Collection of Czechoslovak Chemical Communications [*Eng., Fr., Ger., Russ.*]
Commis. Energ. At. [*Fr.*], *Rapp.*	Commissariat à l'Energie Atomique [France], Rapport [*Fr.*]
Commun. Soil Sci. Plant Anal.	Communications in Soil Science and Plant Analysis [*Eng.*]
C. R. Hebd. Seances Acad. Sci.	Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences [*Fr.*]
Ser. C	Série C: Sciences Chimiques
Ser. D	Série D: Sciences Naturelles
Crit. Rev. Anal. Chem.	Critical Reviews in Analytical Chemistry [*Eng.*]
Croat. Chem. Acta	Croatica Chemica Acta [*Eng., Fr., Ger., Russ.*]
C. R. Seances Acad. Agric. Fr.	Comptes Rendus des Séances de l'Académie d'Agriculture de France [*Fr.*]
Curr. Awareness Part. Technol.	Current Awareness in Particle Technology [*Eng.*]

Curr. Sci.	Current Science [*Eng.*]
Dokl. Akad. Nauk Arm. SSR	Doklady Akademii Nauk Armyanskoi SSR [*Arm., Russ.*]
Dokl. Akad. Nauk SSSR	Doklady Akademii Nauk SSSR [*Russ.*]
Dokl. Akad. Nauk Uzb. SSR	Doklady Akademii Nauk Uzbekskoi SSR [*Russ.*]
Dokl. Bolg. Akad. Nauk	Doklady Bolgarskoi Akademii Nauk [*Eng., Fr., Ger., Russ.*]
Double Liaison - Chim. Peint.	Double Liaison Chimie des Peintures [*Fr.*]
Dtsch. Apoth.-Ztg.	Deutsche Apotheker-Zeitung [*Ger.*]
Dtsch. Lebensm.-Rundsch.	Deutsche Lebensmittel-Rundschau [*Ger.*]
Eczacilik Bul.	Eczacilik Bulteni [*Eng., Turk.*]
Educ. Chem.	Education in Chemistry [*Eng.*]
Eisei Kagaku	Eisei Kagaku (Journal of Hygienic Chemistry) [*Jap.*]
Energy World	Energy World [*Eng.*]
Environ. Sci. Technol.	Environmental Science and Technology [*Eng.*]
**ERDA Energy Res. Abstr.*	ERDA Energy Research Abstracts [*Eng.*]
Erdoel Kohle, Erdgas, Petrochem. Brennst.-Chem.	Erdöl und Kohle, Erdgas, Petrochemie vereinigt mit Brennstoff-Chemie [*Eng., Ger.*]
Essenze Deriv. Agrum.	Essenze e Derivati Agrumari [*Ital.*]
**Euro Abstr.*	Euro Abstracts [*Eng., Fr., Ger., Ital.*]
Experientia	Experientia [*Eng., Fr., Ger.*]
Farbe Lack	Farbe + Lack [*Ger.*]
Farmaco, Ed. Prat.	Farmaco, Edizione Pratica [*Eng., Fr., Ital.*]
Farmaco, Ed. Sci.	Farmaco, Edizione Scientifica [*Eng., Fr., Ital.*]
Farmatsiya (Moscow)	Farmatsiya. Moscow [*Russ.*]
Farm. Obz.	Farmaceuticky Obzor [*Czech, Slovak*]
Farm. Pol.	Farmacja Polska [*Pol.*]
Faserforsch. Textiltech.	Faserforschung und Textiltechnik—Zeitschrift für Polymerforschung [*Ger.*]
Fert. Technol.	Fertilizer Technology (*formerly* Technology) [*Eng.*]
Fette, Seifen, Anstrichm.	Fette, Seifen, Anstrichmittel [*Eng., Ger.*]
Finn. Chem. Lett.	Finnish Chemical Letters [*Eng., Fr., Ger.*]
Fleischwirtschaft	Fleischwirtschaft [*Eng., Ger.*]
Food Chem.	Food Chemistry [*Eng.*]
**Food R.A. Abstr.*	Food R.A. Abstracts [*Eng.*]
Forensic Sci.	Forensic Science [*Eng., Fr., Ger.*]
Fresenius' Z. Anal. Chem.	Fresenius' Zeitschrift für Analytische Chemie [*Eng., Ger.*]
**Fuel Abstr. Curr. Titles*	Fuel Abstracts and Current Titles [*Eng.*]
G.B., Warren Spring Lab., Rep.	Great Britain, Warren Spring Laboratory, Report [*Eng.*]
Geochim. Cosmochim. Acta	Geochimica et Cosmochimica Acta [*Eng.*]
Geokhimiya	Geokhimiya [*Russ.*]
GIT Fachz. Lab.	GIT Fachzeitschrift für das Laboratorium [*Ger.*]
Gordian	Gordian [*Eng., Ger.*]
Grasas Aceites (Seville)	Grasas y Aceites [*Span.*]
Health Phys.	Health Physics [*Eng.*]
Helv. Chim. Acta	Helvetica Chimica Acta [*Fr., Ger.*]
Herba Pol.	Herba Polonica [*Pol.*]
Hoppe-Seyler's Z. Physiol. Chem.	Hoppe-Seyler's Zeitschrift für Physiologische Chemie [*Ger.*]
Hua Hsueh Tung Pao	Huaxue Tongbao (Chemistry) [*Chin.*]
**Hung. Tech. Abstr.*	Hungarian Technical Abstracts [*Eng.*]
Hutn. Listy	Hutnicke Listy [*Czech*]
Ind. Agrar.	Industrie Agrarie [*Ital.*]
Ind. Aliment. Agric.	Industries Alimentaires et Agricoles [*Fr.*]
Indian J. Appl. Chem.	Indian Journal of Applied Chemistry [*Eng.*]
Indian J. Biochem. Biophys.	Indian Journal of Biochemistry & Biophysics [*Eng.*]
Indian J. Chem.	Indian Journal of Chemistry [*Eng.*]
Sect. A	Section A: Inorganic, Physical, Theoretical & Analytical
Sect. B	Section B: Organic including Medicinal
Indian J. Pharm.	Indian Journal of Pharmacy [*Eng.*]
Indian J. Pure Appl. Phys.	Indian Journal of Pure and Applied Physics [*Eng.*]
Indian J. Technol.	Indian Journal of Technology [*Eng.*]
**INIS Atomindex*	INIS Atomindex [*Eng.*]
Instrum. Control Syst.	Instruments and Control Systems [*Eng.*]
Int. Flavours Food Addit.	International Flavours and Food Additives [*Eng.*]
Int. J. Appl. Radiat. Isot.	International Journal of Applied Radiation and Isotopes [*Eng., Fr., Ger., Russ.*]
Int. J. Mass Spectrom. Ion Phys.	International Journal of Mass Spectrometry and Ion Physics [*Eng., Fr., Ger.*]
Int. Lab.	International Laboratory [*Eng.*]
Int. Sugar J.	International Sugar Journal [*Eng.*]
Isotopenpraxis	Isotopenpraxis [*Eng., Ger., Russ.*]
Isr. J. Chem.	Israel Journal of Chemistry [*Eng.*]
Ital. J. Biochem.	Italian Journal of Biochemistry [*Eng.*]
Izv. Akad. Nauk Kaz. SSR, Ser. Khim.	Izvestiya Akademii Nauk Kazakhskoi SSR, Seriya Khimicheskaya [*Russ.*]

Izv. Akad. Nauk SSSR, Ser. Khim.	Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya [*Russ.*]
Izv. Tomsk. Politekh. Inst.	Izvestiya Tomskogo Politekhnicheskogo Instituta [*Russ.*]
Izv. Vyssh. Uchebn. Zaved., Khim. Khim. Tekhnol.	Izvestiya Vysshikh Uchebnykh Zavedenii, Khimiya i Khimicheskaya Tekhnologiya [*Russ.*]
J. Aerosol Sci.	Journal of Aerosol Science [*Eng., Fr., Ger.*]
J. Agric. Food Chem.	Journal of Agricultural and Food Chemistry [*Eng.*]
J. Air Pollut. Control Assoc. ..	Journal of the Air Pollution Control Association [*Eng.*]
J., Am. Ind. Hyg. Assoc.	Journal, American Industrial Hygiene Association (*Eng.*]
J. Am. Oil Chem. Soc.	Journal of the American Oil Chemists' Society [*Eng.*]
J. Am. Pharm. Assoc.	Journal of the American Pharmaceutical Association [*Eng.*]
J. Am. Water Works Assoc. ..	Journal of the American Water Works Association [*Eng.*]
J. Appl. Chem. Biotechnol.	Journal of Applied Chemistry & Biotechnology [*Eng.*]
J. Appl. Polym. Sci.	Journal of Applied Polymer Science [*Eng.*]
J. Assoc. Off. Anal. Chem.	Journal of the Association of Official Analytical Chemists [*Eng.*]
J. Assoc. Public Anal.	Journal of the Association of Public Analysts [*Eng.*]
J. Chem. Educ.	Journal of Chemical Education [*Eng.*]
J. Chem. Res.	Journal of Chemical Research [*Eng., Fr., Ger.*]
J. Chem. Soc., Chem. Commun. ..	Journal of the Chemical Society, Chemical Communications [*Eng.*]
J. Chem. Soc., Perkin Trans. 1 ..	Journal of the Chemical Society, Perkin Transactions I [*Eng.*]
J. Chem. Soc., Perkin Trans. 2 ..	Journal of the Chemical Society, Perkin Transactions II [*Eng.*]
J. Chim. Phys. Phys.-Chim. Biol. ..	Journal de Chimie Physique et de Physico-Chimie Biologique [*Fr.*]
J. Chin. Chem. Soc. (Taipei) ..	Journal of the Chinese Chemical Society. Taipei [*Eng.*]
J. Chromatogr.	Journal of Chromatography [*Eng., Fr., Ger.*]
J. Chromatogr. Sci.	Journal of Chromatographic Science [*Eng.*]
J. Clin. Chem. Clin. Biochem. ..	Journal of Clinical Chemistry and Clinical Biochemistry [*Eng., Ger.*]
J. Coat. Technol.	Journal of Coatings Technology (*formerly* Journal of Paint Technology) [*Eng.*]
J. Colloid Interface Sci.	Journal of Colloid and Interface Science [*Eng.*]
J. Dairy Res.	Journal of Dairy Research [*Eng.*]
J. Dairy Sci.	Journal of Dairy Science [*Eng.*]
J. Drug Res.	Journal of Drug Research [*Eng.*]
J. Electroanal. Chem. Interfacial Electrochem.	Journal of Electroanalytical Chemistry and Interfacial Electrochemistry [*Eng., Fr., Ger.*]
J. Electrochem. Soc.	Journal of the Electrochemical Society [*Eng.*]
Jena Rev.	Jena Review [*Eng.*]
J. Environ. Sci. Health	Journal of Environmental Science and Health (*formerly* Environmental Letters) [*Eng.*]
Part A	Part A: Environmental Science and Engineering
Part B	Part B: Pesticides, Food Contaminants, and Agricultural Wastes
J. Food Sci.	Journal of Food Science [*Eng.*]
J. Food Sci. Technol.	Journal of Food Science and Technology [*Eng.*]
J., Forensic Sci. Soc.	Journal, Forensic Science Society [*Eng.*]
J. Gen. Chem. USSR (Engl. Transl.)	Journal of General Chemistry of the USSR [*Eng.*]
J. Indian Chem. Soc.	Journal of the Indian Chemical Society [*Eng.*]
J. Inorg. Nucl. Chem.	Journal of Inorganic and Nuclear Chemistry [*Eng.*]
J. Inst. Brew., London	Journal of the Institute of Brewing, London [*Eng.*]
J. Inst. Fuel	Journal of the Institute of Fuel [*Eng.*]
J. Lab. Clin. Med.	Journal of Laboratory and Clinical Medicine [*Eng.*]
J. Labelled Compd. Radiopharm. ..	Journal of Labelled Compounds and Radiopharmaceuticals [*Eng., Fr., Ger.*]
J. Lipid Res.	Journal of Lipid Research [*Eng.*]
J. Nutr. Sci. Vitaminol.	Journal of Nutritional Science and Vitaminology [*Eng.*]
J. Pharm. Belg.	Journal de Pharmacie de Belgique [*Fr.*]
J. Pharm. Pharmacol.	Journal of Pharmacy and Pharmacology [*Eng.*]
J. Pharm. Sci.	Journal of Pharmaceutical Sciences [*Eng.*]
J. Phys. D	Journal of Physics D: Applied Physics [*Eng.*]
J. Phys. E	Journal of Physics E: Scientific Instruments [*Eng., Fr., Ger.*]
J. Polym. Sci., Polym. Chem. Ed. ..	Journal of Polymer Science, Polymer Chemistry Edition [*Eng., Fr., Ger.*]
J. Prakt. Chem.	Journal für Praktische Chemie [*Eng., Ger.*]
J. Radioanal. Chem.	Journal of Radioanalytical Chemistry [*Eng., Fr., Ger.*]
J. Res. Natl. Bur. Stand., Sect. A ..	Journal of Research of the National Bureau of Standards, Section A: Physics and Chemistry [*Eng.*]
J. S. Afr. Inst. Min. Metall. ..	Journal of the South African Institute of Mining & Metallurgy [*Eng.*]
J. Sci. Food Agric.	Journal of the Science of Food and Agriculture [*Eng.*]

J. Sci. Ind. Res.	Journal of Scientific and Industrial Research [*Eng.*]
J. Soc. Leather Technol. Chem.	Journal of the Society of Leather Technologists and Chemists [*Eng.*]
J. Steroid Biochem.	Journal of Steroid Biochemistry [*Eng., Fr., Ger.*]
J. Water Pollut. Control Fed.	Journal of the Water Pollution Control Federation [*Eng.*]
Kautsch. Gummi, Kunstst.	Kautschuk und Gummi, Kunststoffe [*Ger.*]
Khim. Ind. (Sofia)	Khimiya i Industriya [*Bulg.*]
Kinet. Katal.	Kinetika i Kataliz [*Russ.*]
Klin. Wochenschr.	Klinische Wochenschrift [*Ger.*]
Kunststoffe	Kunststoffe [*Ger.*]
Lab. Delo	Laboratornoe Delo [*Russ.*]
Lab. Pract.	Laboratory Practice [*Eng.*]
Landwirtsch. Forsch.	Landwirtschaftliche Forschung [*Ger.*]
Latv. PSR Zinat. Akad. Vestis, Kim. Ser.	Latvijas PSR Zinatnu Akademijas Vestis, Kimijas Serija [*Russ.*]
**Lead Abstr.*	Lead Abstracts [*Eng.*]
Lebensm.-Wiss. Technol.	Lebensmittel-Wissenschaft + Technologie [*Eng., Fr., Ger.*]
Lichtbogen	Lichtbogen [*Eng., Fr., Ger.*]
Lipids	Lipids [*Eng.*]
Lloydia	Lloydia [*Eng.*]
Magy. Kem. Foly.	Magyar Kemiai Folyoirat [*Hung.*]
Magy. Kem. Lapja	Magyar Kemikusok Lapja [*Hung.*]
Medd. Nor. Farm. Selsk.	Meddelelser fra Norsk Farmaceutisk Selskap [*Eng.*]
Med. Lab. Sci.	Medical Laboratory Sciences (*formerly* Medical Laboratory Technology) [*Eng.*]
Med., Sci. Law	Medicine, Science and the Law [*Eng.*]
Melliand Textilber. Int.	Melliand Textilberichte International [*Ger.*]
Metall. Ital.	Metallurgia Italiana [*Ital.*]
Metall. Met. Form.	Metallurgia and Metal Forming [*Eng.*]
Met. Sci.	Metal Science [*Eng.*]
Met. Technol. (London)	Metals Technology [*Eng.*]
Microchem. J.	Microchemical Journal [*Eng.*]
Mikrochim. Acta	Mikrochimica Acta [*Eng., Fr., Ger.*]
**Mineral. Abstr.*	Mineralogical Abstracts [*Eng.*]
Mitt. Geb. Lebensmittelunters. Hyg.	Mitteilungen aus dem Gebiete der Lebensmitteluntersuchung und Hygiene [*Fr., Ger.*]
Mol. Cell. Biochem.	Molecular and Cellular Biochemistry [*Eng.*]
Monatsh. Chem.	Monatshefte für Chemie [*Eng., Ger.*]
Monatsschr. Brau.	Monatsschrift für Brauerei [*Ger.*]
Nahrung	Nahrung [*Eng., Ger., Russ.*]
Natl. Bur. Stand. (U.S.), Spec. Publ.	National Bureau of Standards (U.S.), Special Publication [*Eng.*]
Natl. Inst. Metall., Repub. S. Afr., Rep.	National Institute of Metallurgy, Republic of South Africa, Report [*Eng.*]
Nature (London)	Nature [*Eng.*]
Notes Water Res.	Notes on Water Research [*Eng.*]
Nucl. Instrum. Methods	Nuclear Instruments & Methods [*Eng., Fr., Ger.*]
**Nutr. Abstr. Rev.*	Nutrition Abstracts and Reviews [*Eng.*]
Paint Manuf.	Paint Manufacture [*Eng.*]
Period. Polytech., Chem. Eng.	Periodica Polytechnica, Chemical Engineering [*Eng., Ger., Russ.*]
Pestic. Sci.	Pesticide Science [*Eng.*]
Pharm. Acta Helv.	Pharmaceutica Acta Helvetiae [*Eng., Fr., Ger.*]
Pharma Int., Engl. Ed.	Pharma International, English Edition [*Eng.*]
Pharmakon	Pharmakon [*Flem., Fr.*]
Pharmazie	Pharmazie [*Eng., Ger.*]
Pharm. Prax.	Pharmaceutische Praxis [*Ger.*]
Pharm. Weekbl.	Pharmaceutisch Weekblad [*Dutch, Eng., Ger.*]
Plaste Kautsch.	Plaste und Kautschuk [*Ger.*]
Pol. J. Pharmacol. Pharm.	Polish Journal of Pharmacology and Pharmacy [*Eng.*]
Polym., Paint Colour J.	Polymers, Paint and Colour Journal [*Eng.*]
Powder Technol.	Powder Technology [*Eng., Fr.*]
Proc. Anal. Div. Chem. Soc.	Proceedings of the Analytical Division of the Chemical Society [*Eng.*]
Proc. K. Ned. Akad. Wet.	Proceedings of the Koninklijke Nederlandse Akademie van Wetenschappen [*Eng.*]
Ser. B	Series B: Physical Sciences
Ser. C	Series C: Biological and Medical Sciences
Prostaglandins	Prostaglandins [*Eng.*]
Prum. Potravin	Prumysl Potravin [*Czech, Slovak*]
Pure Appl. Chem.	Pure and Applied Chemistry [*Eng., Fr.*]
Pyrethrum Post	Pyrethrum Post [*Eng., Fr., Ger.*]
Quim. Anal.	Quimica Analitica [*Span.*]
Radiochem. Radioanal. Lett.	Radiochemical and Radioanalytical Letters [*Eng., Fr., Ger.*]

**RAPRA Abstr.*	RAPRA Abstracts [*Eng.*]
Ref. Zh., Biol. Khim.	Referativnyi Zhurnal, Biologicheskaya Khimiya [*Russ.*]
Ref. Zh., Khim., 19GD	Referativnyi Zhurnal, Khimiya. Sections G and D [*Russ.*]
Ref. Zh., Khim., 19R	Referativnyi Zhurnal, Khimiya. Section R [*Russ.*]
Res./Dev.	Research/Development [*Eng.*]
Rev. Asoc. Bioquim. Argent.	Revista de la Asociacion Bioquimica Argentina [*Span.*]
Rev. Bras. Tecnol.	Revista Brasileira de Tecnologia [*Port.*]
Rev. CENIC, Cienc. Biol.	Revista CENIC, Ciencias Biologicas [*Span.*]
Rev. CENIC, Cienc. Fis.	Revista CENIC, Ciencias Fisicas [*Span.*]
Rev. Chim. (Bucharest)	Revista de Chimie [*Rom.*]
Rev. Farm. Bioquim. Univ. Sao Paulo	Revista de Farmacia e Bioquimica da Universidade de Sao Paulo [*Port.*]
Rev. Metal. (Madrid)	Revista de Metalurgia [*Span.*]
Rev. Pol. Acad. Sci.	Review of the Polish Academy of Sciences [*Eng.*]
Rev. Roum. Biochim.	Revue Roumaine de Biochimie [*Eng., Fr., Ger., Russ.*]
Rev. Roum. Chim.	Revue Roumaine de Chimie [*Eng., Fr., Ger., Russ.*]
Rev. Sci. Instrum.	Review of Scientific Instruments [*Eng.*]
Riechst., Aromen, Kosmet.	Riechstoffe, Aromen, Kosmetica (*formerly* Riechstoffe, Aromen, Körperpflegemittel) [*Ger.*]
Riv. Ital. Sostanze Grasse	Rivista Italiana delle Sostanze Grasse [*Ital.*]
Rocz. Chem.	Roczniki Chemii [*Eng., Pol.*]
Rocz. Panstw. Zakl. Hig.	Roczniki Panstwowego Zakladu Higieny [*Pol.*]
Scand. J. Metall.	Scandinavian Journal of Metallurgy [*Eng.*]
Science	Science [*Eng.*]
Sci. Light (Tokyo)	Science of Light [*Eng.*]
Sci. Pharm.	Scientia Pharmaceutica [*Ger.*]
Sci. Tools	Science Tools [*Eng.*]
Scr. Fac. Sci. Nat. Univ. Purkynianae Brun.	Scripta Facultatis Scientiarum Naturalium Universitatis Purkynianae Brunensis [*Czech, Eng., Ger., Russ.*]
Ser. Biol.	Series Biologia
Ser. Chem.	Series Chemia
Ser. Phys.	Series Physica
Sep. Purif. Methods	Separation and Purification Methods [*Eng.*]
Sep. Sci.	Separation Science [*Eng.*]
Spectra 2000	Spectra 2000 [*Eng., Fr.*]
Spectrochim. Acta	Spectrochimica Acta [*Eng., Fr., Ger.*]
Part A	Part A: Molecular Spectroscopy
Part B	Part B: Atomic Spectroscopy
Spectrosc. Lett.	Spectroscopy Letters [*Eng.*]
Staub–Reinhalt. Luft	Staub–Reinhaltung der Luft [*Ger.*]
Steroids	Steroids [*Eng.*]
Stud. Univ. Babes-Bolyai, Ser. Chem.	Studia Universitatis Babes-Bolyai, Series Chemia [*Eng., Fr., Ger., Rom.*]
Swed. J. Agric. Res.	Swedish Journal of Agricultural Research [*Eng., Fr., Ger.*]
Talanta	Talanta [*Eng., Fr., Ger.*]
Tappi	Tappi [*Eng.*]
Tecnica (Lisbon)	Tecnica [*Eng., Port.*]
Trav. Soc. Pharm. Montpellier	Travaux de la Société de Pharmacie de Montpellier [*Fr.*]
Trib. CEBEDEAU	Tribune de CEBEDEAU [*Fr.*]
Trop. Sci.	Tropical Science [*Eng.*]
Turk Hij. Den. Biyol. Derg.	Turk Hijiyen ve Deneysel Biyoloji Dergisi (*formerly* Turk Hijiyen ve Tecrubi Biyoloji Dergisi) [*Eng., Turk.*]
Uch. Zap., Permsk. Gos. Univ.	Uchenye Zapiski, Permskii Gosudarstvennyi Universitet [*Russ.*]
U.K. At. Energy Res. Establ., Rep.	United Kingdom Atomic Energy Research Establishment, Report [*Eng.*]
Ukr. Khim. Zh. (Russ. Ed.)	Ukrainskii Khimicheskii Zhurnal [*Russ.*]
U.S., Bur. Mines, Rep. Invest.	United States, Bureau of Mines, Report of Investigations [*Eng.*]
U.S. Energy Res. Dev. Adm., Rep.	United States Energy Research and Development Administration, Report [*Eng.*]
UV Spectrom. Group Bull.	UV Spectrometry Group Bulletin [*Eng.*]
Uzb. Khim. Zh.	Uzbekskii Khimicheskii Zhurnal [*Russ.*]
Vestn. Leningr. Univ., Fiz., Khim.	Vestnik Leningradskogo Universiteta, Fizika, Khimiya [*Russ.*]
Vestn. Mosk. Univ., Khim.	Vestnik Moskovskogo Universiteta, Khimiya [*Russ.*]
Water Res.	Water Research [*Eng.*]
Water Sewage Works	Water & Sewage Works [*Eng.*]
**World Surf. Coat. Abstr.*	World Surface and Coatings Abstracts [*Eng.*]
**World Text. Abstr.*	World Textile Abstracts [*Eng.*]
**WRC Inf.*	WRC Information [*Eng.*]
X-Ray Spectrom.	X-Ray Spectrometry [*Eng., Fr., Ger.*]
Yakugaku Zasshi	Yakugaku Zasshi (Journal of the Pharmaceutical Society of Japan) [*Jap.*]

Z. Anorg. Allg. Chem.	Zeitschrift für Anorganische und Allgemeine Chemie [*Eng., Ger.*]
Zavod. Lab.	Zavodskaya Laboratoriya [*Russ.*]
Z. Chem.	Zeitschrift für Chemie [*Ger.*]
Zem.-Kalk-Gips	Zement-Kalk-Gips [*Ger.*]
Zentralbl. Pharm., Pharmakother. Laboratoriumsdiagn.	Zentralblatt für Pharmazie, Pharmakotherapie und Laboratoriumsdiagnostik [*Ger.*]
Zh. Anal. Khim.	Zhurnal Analiticheskoi Khimii [*Russ.*]
Zh. Obshch. Khim.	Zhurnal Obshchei Khimii [*Russ.*]
Zh. Prikl. Khim. (Leningrad) ..	Zhurnal Prikladnoi Khimii. Leningrad [*Russ.*]
Zh. Prikl. Spektrosh.	Zhurnal Prikladnoi Spektroskopii [*Russ.*]
Zh. Strukt. Khim.	Zhurnal Strukturnoi Khimii [*Russ.*]
Zh. Vses. Khim. O-va	Zhurnal Vsesoyuznogo Khimicheskogo Obshchestva im. D. I. Mendeleeva [*Russ.*]
**Zinc Abstr.*	Zinc Abstracts [*Eng.*]
Z. Lebensm.-Unters. Forsch. ..	Zeitschrift für Lebensmittel-Untersuchung und -Forschung [*Eng., Ger.*]
Z. Naturforsch., Teil C	Zeitschrift für Naturforschung, Teil C: Biosciences [*Eng., Ger.*]

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Analytical Abstracts

A—GENERAL ANALYTICAL CHEMISTRY

Proceedings of conferences; general reviews of progress; reagents; methods of general application; hazards

1A1. **Spectrophotometric methods of analysis of high-purity substances.** Nazarenko, V. A.; and Flyantikova, G. V. (Inst. Gen. and Inorg. Chem., Acad. Sci. Ukr. SSR, Odessa). *Zh. Anal. Khim.*, 1977, **32** (6), 1217–1236 (in Russian).—Methods for determining traces of ≃40 impurity elements in acids, hydroxides, salts, metals and alloys are reviewed. (294 references.) M. P.

1A2. **Numerical consideration of mutual effects of two components, illustrated by flame-photometric determination of sodium and potassium.** Volke, Peter (Forschungsinst. Aufbereit., D.A.W., Freiberg, E. Germany). *Silikattechnik*, 1976, **27** (4), 130–132 (in German).—The mutual interference of Na and K during such determination has been evaluated by use of sets of calibration graphs (for up to 100 mg l^{-1} of Na_2O or K_2O) and a programmable calculator. An iteration program containing two equations for the calculation of Na and K concn. takes into account this interference, and a procedure is described for obtaining the equations from measurement points of the calibration-graph sets. The equation for each graph is determined by polynomial curve fitting. The efficacy of the equations for use when up to 1 g l^{-1} of K_2O and up to 100 mg l^{-1} of Na_2O are present is established. CHEM. ABSTR.

1A3. **Analytical applications of 2-hydroxymercuribenzoic acid, dithiofluorescein, and mercurated fluorescein.** Wronski, Mieczyslaw (Dept. Chem./Petro-Chem. Eng., Coll. Sci. and Technol., Port Harcourt, Nigeria). *Talanta*, 1977, **24** (6), 347–354.—2-Hydroxymercuribenzoic acid (**I**) is superior to other compounds of Hg^{II} as a reagent for H_2S and thiols and may be used in both alkaline and acid media; it accelerates the alkaline hydrolysis of S compounds. Dithiofluorescein (**II**) and **I** form a colourless complex, which is decomposed by thiols to give the blue **II**. Mercurated fluorescein in alkaline soln. gives a green fluorescence, which is quenched by several S and N compounds. The application of these reagents to titrimetric, photometric and fluorimetric analysis of compounds of S, N and Hg, including procedures for selective determinations, is reviewed. (132 references.) T. J. S.

1A4. **Analytical reactions of hydroxyflavones.** Katyal, Mohan; and Prakash, Shri (St. Stephen's Coll., Delhi, India). *Talanta*, 1977, **24** (6), 367–375.—In continuation of an earlier survey (*Anal. Abstr.*, 1969, **16**, 1736), recent applications of hydroxyflavones, *e.g.*, morin, quercetin, rutin, kaempferol and flavonol, as reagents for metals and various anions are reviewed. (83 references.) T. J. S.

1A5. **5,8-Dihydroxyanthraquinone-2,3-dicarboxylic acid as analytical reagent for metal ions.** Dubrovina, L. T.; Luginin, V. A.; Tserkovnitskaya, I. A.; Kalacheva, N. I.; and Medvedev, Yu. V. *Probl. Sovrem. Anal. Khim.*, 1976, **1**, 123–131 (in Russian).—The named reagent (prepared from pyromellitic dianhydride and hydroquinone in the presence of H_2SO_4 and H_3BO_3 at 150°) forms, at pH 4·2 to 6·2, complexes with Be, Al, V^{IV}, U^{VI} and Mo^{VI} having absorption max. at 575, 565, 570, 610 and 560 nm, respectively, the corresponding ϵ values being 3500, 4250, 4650, 2370 and 4560; addition of ethanol or dimethylformamide increases the solubility of these complexes. Tervalent Cr, La and Th also form coloured complexes with this reagent. CHEM. ABSTR.

1A6. **Synthesis and analytical investigation of azo-compounds based on tetrahydro-1,3-thiazine-2,4-dithione.** Rozovskii, Yu. G.; and Basargin, N. N. (Inst. Ore Deposits, Petrogr., Mineral. and Geochem., Acad. Sci. USSR). *Zh. Vses. Khim. O-va.*, 1977, **22** (4), 467–468 (in Russian).—Nine complexes based on this compound and various substituted 2-aminophenols were examined as reagents for spectrophotometric determination of Ag, Au, Pt, Pd and Cu. Data on the solubility, stability and absorption max. of the complexes are given. M. R. M.

1A7. **Sample preparation by 'chemical masking'.** Chamberlain, J. (Hoechst Pharm. Res. Lab., Milton Keynes, Bucks., England). *Methodol. Dev. Biochem.*, 1976, **5**, 125–130.—This technique (in which the compound to be determined is not separated from interfering species, but the test soln. is so treated as to permit determination of only that compound) is briefly discussed, and examples are given of its use. These include—(*i*) in assays involving u.v. spectrophotometry, prevention of interference from barbiturates or phenols by changing the pH; (*ii*) reduction of the 3-oxo-group of progesterone, so eliminating its absorption at 240 nm; (*iii*) oxidation of urinary steroids to form a compound amenable to electron-capture detection in g.l.c.; and (*iv*) use of salicylaldehyde to form Schiff bases with primary amines and prevent their interference when determining, *e.g.*, secondary amines. B. H.

1A8. **Oxidimetric determination of some compounds with hexa-amminecobalt(III) tricarbonatocobaltate(III).** Vasatova, Miloslava; and Zyka, Jaroslav (Dept. Anal. Chem., Charles Univ., Prague, Czechoslovakia). *Microchem. J.*, 1977, **22** (1), 34–44.—A 5mN soln. of the reagent (prep. indicated) at pH 7·6 was used as titrant for the potentiometric determination of I^-, ascorbic acid, SCN^-, $S_2O_3^{2-}$ and H_2O_2, with use of a platinum-wire indicator electrode

and a s.c.e. Direct and indirect titrations were performed. The effects of Cl^-, Br^- and NO_3^- on the determination of I^- are discussed. T. G. A.

1A9. **Recent applications of digital computers in analytical chemistry.** Perrin, D. D. (Med. Chem. Group, John Curtin Sch. Med. Res., Australian Natl. Univ., Canberra). *Talanta*, 1977, **24** (6), 339–345.—Uses of mini-computers and of dedicated microcomputers in analytical chemistry are reviewed. Applications of computers in electroanalytical chemistry, Fourier-transform techniques, spectroscopy, rapid-reaction kinetics, the calculation of equilibrium constants, theoretical studies of analytical methods and literature searching are discussed. (152 references.) T. J. S.

See also Abstracts: **1B8**, Petroleum-based extractants for metals. **1B10**, Reagent for chemical microscopy. **1B13**, Complex of Cu for coulometric determination of metal ions. **1B15**, Fluorimetric reagent for various metals. **1B16**, Chelating reagent for separation of metals by g.l.c. **1B24**, Indicator for complexometric titrations of metal ions. **1B31**, **1B32**, Reagents for Cu. **1B34**, Reagents for Cu, Ag and Pd. **1B35**, Reagent for Cu and Ni. **1B37**, Extractant for Cu. **1B44**, Reagent for Ag. **1B48**, Reagent for Au^{III}. **1B52**, Reagent for alkaline-earth metals and lanthanoids. **1B55**, Reagent system for Mg and Fe. **1B56**, Comparison of indicators for titration of Ca. **1B61**, Reagent for separation of Sr from Ca. **1B62**, Reagent for Ba. **1B66**, Reagents for metals of the Zn sub-group. **1B67**, Reagent system for Zn, Co or Ni. **1B70**, **1D15**, Extractants for Cd. **1B73**, Study of extraction of Hg. **1B76**, Reagents for Al. **1B80**, **1B81**, Reagent systems for Ga. **1B84**, Reagent for In. **1B85**, Titrant for In and Cu. **1B86**, Reagent system for Tl. **1B91**, Precipitant for Ce. **1B92**, Reagent for Ce. **1B93**, Reagent systems for Nd, Ho and Er. **1B104**, Indicators for edta titration of Th and Zr. **1B120**, Reagent system for Ge. **1B127**, **1B154** to **1B156**, Reagent for Bi. **1B128** to **1B131**, Reagent systems for Ti. **1B135** to **1B137**, Reagents for Zr. **1B138**, Reagent for Zr and Hf. **1B141**, Reagent for NO_3^-. **1B146**, Determination of PO_4^{3-} in hydrochloric and hydrobromic acids. **1B148**, Indicators for titration of As^{III}. **1B152**, Reagent for Sb. **1B158**, **1B159**, Reagent systems for V. **1B163**, Extraction system for Nb. **1B177**, Reagent system for W. **1B184**, Titrants for ClO_4^-. **1B185**, Extraction constants for HBr, HI and HSCN. **1B194**, Extraction system for Fe. **1B210**, Reagent for Co, Ni and Cu. **1B218**, Reagent for Rh. **1B219**, **1B221**, Reagents for Pd. **1B220**, Indicators for photometric titration of Pd. **1B226**, Reagents for Os. **1C38**, Indicators for titration of mercaptoacetic acid. **1C48**, Reagent for chloramine T. **1D7**, Computer program for g.c. of cigarette smoke. **1D23**, Reagents for g.c. determination of Se. **1D138**, Reagent for amino-compounds. **1E9**, Reagents for Fe and Co. **1E12**, Reagents for various drugs: review. **1E43**, Reagent for use in analysis of antibiotics. **1E60**, **1E62**, **1E63**, Reagents for determination of drugs. **1E67**, Spray reagent for t.l.c. of corticosteroids. **1F3**, Trace analysis for Cd, Pb, Hg and methylmercury: review. **1G9**, Reagent for B. **1H37**, Reagent for NH_3. **1H51**, Reagents for phenolic compounds. **1H55**, Reagent for Hg. **1J10**, Computerisation of AutoAnalyzers. **1J13**, Immobilised reagents for sorption of metals from water. **1J46**, Micro-computer system for g.c. **1J49**, Colour techniques in electron microscopy: review. **1J51**, Optical emission spectrometry: review. **1J60**, Mathematical evaluation of errors in X-ray analysis. **1J89**, Computer program for interpretation of i.r. and Raman spectra. **1J99**, Determinations with use of two radionuclides: review. **1J101**, Computerised correction in liquid scintillation counting.

B—INORGANIC CHEMISTRY

General

1B1. **Reduction of the detection limits for micro amounts [of elements] in spectrographic analysis of high-purity products.** Sabatovskaya, V. L.; Kuzovlev, I. A.; and Yudelevich, I. G. *Zh. Prikl. Spektrosk.*, 1977, **26** (2), 207–212 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G208.

1B2. **Determination of concentrations of metal atoms from changes in the relative intensities of ultra-fine-structure spectral lines.** Zhiglinskii, A. G.; Kuchinskii, V. V.; Milovanov, N. P.; and Presnukhina, I. P. *Opt. Spektrosk.*, 1977, **42** (3), 427–430 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (17), Abstr. No. 17G36.

1B3. **Atomic-absorption determination of several elements by atomisation of the sample in an arc discharge.** Dorofeev, V. S.; Mikhailov, N. S.; Razyapov, A. Z.; and Chupakhin, M. S. *Zh. Prikl. Spektrosk.*, 1977, **26** (4), 589–592 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (16), Abstr. No. 16G26.

1B4. **Analysis of real errors, and means for their reduction, in precise determination of isotopes.** Doil'nitsyn, E. F.; Zyuzin, V. I.; and Pertseva, A. P. *Tr. Inst. Geol. Geofiz. Akad. Nauk SSR, Sib. Otd.*, 1977, (315), 49–53 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (16), Abstr. No. 16G43.

1B5. **Determination of forty elements in geochemical samples and coal fly ash by X-ray fluorescence spectrometry.** Giauque, Robert D.; Garrett, Roberta B.; and Goda, Lilly Y. (Energy and Environ. Div., Lawrence Berkeley Lab., Univ. California, Berkeley, Calif. 94720, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1012–1017.—The finely powdered sample (100 mg) is mixed with 400 mg of S, and the mixture is compressed at 15,000 p.s.i. to form a 1-in. disc, which is examined by X-ray fluorescence in an instrument having a low-power X-ray tube (tungsten anode) and interchangeable secondary targets (molybdenum and terbium). The S provides a substantially similar matrix for different sample materials, thus permitting single matrix corrections to be applied. Standardisation is achieved by means of deposition from nebulised soln. and from vapour-deposited single elements. Results are reported for USGS standard andesite, basalt and granite and for NBS coal fly ash SRM 1633. R. W.

1B6. **Radiochemical separations by liquid - liquid chromatography using a porous silica microsphere support.** Horwitz, E. P.; Bloomquist, C. A. A.; and Delphin, W. H. (Chem. Div., Argonne Natl. Lab., Ill. 60439, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (2), 41–46.—Various main-group elements, lanthanoids and transuranium elements have been separated on short columns (20 mm × 2 to 3 mm) packed

with Zorbax-SIL (4 to 6 μm) coated with M-bis-(2-ethylhexyl) hydrogen phosphate in dodecane, with dil. mineral acids as mobile phases. Column performance is evaluated by the elution of Eu^{III}.
M. J. Sa.

1B7. **Rapid separation of metal ions on hydrated zirconium oxide paper.** Singh, N. J.; and Tandon, S. N. (Dept. Chem., Univ. Roorkee, India). *Chromatographia*, 1977, **10** (6), 309–311.—The adsorbent is prepared by treatment of Whatman No. 1 paper with 0·1M-$ZrOCl_2$ and 6% NH_3 soln.; solvent systems include aq. mixtures of HCl, KCl and NH_4Cl. A successful separation of 58 metal ions is achieved within 50 min. M. J. Sa.

1B8. **Study of extraction capability of 'petroleum sulphoxides' by reversed-phase paper chromatography.** Murinov, Yu. I.; Kartavtseva, A. G.; and Nikitin, Yu. E. (Inst. Chem., Acad. Sci. USSR, Bashkir Branch, Ufa). *Zh. Anal. Khim.*, 1977, **32** (5), 904–908 (in Russian).—The possibility of using 'petroleum sulphoxides' prepared by oxidation of a 'petroleum sulphide' fraction (boiling-range 260° to 360°) for the extraction and extraction-chromatographic separation of 50 metals has been studied by paper chromatography. The R_F values of the metals were determined on paper impregnated with a 0·1M soln. of the sulphoxides in light petroleum, with 0·1 to 8M-HCl (or HNO_3), 0·1 to 5M-HNO_3 plus 5 to 0·1M-HCl, or 2·5M-H_2SO_4 that is 0·1 to 6M in NaCl as mobile phase. Separations attainable included those of—Zr from Hf; Pd^{II} from Pt^{IV} and Rh^{III}; Tl^{III} from In and Ga; Te^{IV} from Se^{IV}; and Sn^{IV} from In^{III}, Ga^{III} and Ge. Tervalent Au, Tl^{III}, Ga^{III}, Fe^{III} and Sb^{III} could be selectively extracted from HCl - HNO_3 mixtures, and V^{V}, Au^{III}, Pd^{II}, In^{III}, Tl^{III} and Mo^{VI} from NaCl - H_2SO_4 mixtures. M. P.

1B9. **Use of electrophoresis in analytical chemistry. I. Combined extraction - electrophoretic method for separation of ions.** Luginin, V. A.; and Tserkovnitskaya, I. A. *Probl. Sovrem. Anal. Khim.*, 1976, **1**, 155–162 (in Russian).—A study was made of the separation of metals by chromatography and electrophoresis (with use of 0·1 to 4M-HCl as developing solvent and electrolyte, respectively) on papers impregnated with 5 to 100% of tributyl phosphate (**I**) in toluene. In both methods, separation factors increased with increasing concn. of **I** or HCl. The mobility order of the metals was converse to their extraction order with **I**; thus, Au^{III} remained at the origin in all instances. Typically, electrophoresis permitted better and faster separations than did chromatography; electrophoretic separation factors with 0·1M-HCl and 50% **I** soln. were—U^{VI} - Fe^{III} 1·27, Ni^{II} - Cu 1·12, Fe^{II} - Fe^{III} 1·60, V^{IV} - V^{V} 1·75, Pt^{IV} - Pt^{II} 1·85, and Sn^{II} - Sn^{IV} 1·70.
Chem. Abstr.

1B10. **Extended use of squaric acid [3,4-dihydroxycyclobut-3-ene-1,2-dione] as a reagent in chemical microscopy.** Whitman, V. L.; and Willis, W. F., jun. (Vallecitos Nucl. Center, General Electric Co., Pleasanton, Calif., U.S.A.). *Microscope*, 1977, **25** (1), 1–13.—Squaric acid was tested as a reagent for chemical microscopy with 42 cations; 18 formed complexes that crystallised on standing. The crystal form unique to each cation allowed the detection of sub-microgram amounts of Na, K, Rb, Cs, Mg, Ca, Sr, Ba, Mn, Cu, Ag, Zn, Hg, Al, Sn, Fe, NH_4^+ and UO_2^{2+} by using a polarising microscope. Squaric acid could be used to detect cations in mixtures and for screening tests before applying other, specific, reagents. Chem. Abstr.

1B11. **Analysis of thin films by electron probe. I. Measurement techniques for thickness determination.** Hofer, Wolfgang O. (MPI Plasmaphys., Garching, Muenchen, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 185–196 (in German).—Coatings of Cu and Ag on aluminium and of Al and Mg on copper were studied; suggested methods of obtaining better sensitivity include enhanced electron scattering in the surface, back-scattering from the matrix or the coating, and the use of the bremsstrahlung from the coating. J. W. P.

1B12. **Masking effects in the conductometric titration of Lewis acids with edta in dimethylformamide.** Yoshimuru, Chozo; and Miyamoto, Kiyoshige (Dept. Appl. Chem., Fac. Sci. and Eng., Kinki Univ., Higashi-Osaka-shi, Osaka, Japan). *Bunseki Kagaku*, 1977, **26** (6), 371–375 (in Japanese).—An investigation has been carried out into the use of masking agents to decrease the number of inflection points in the titration of metal chlorides with edta in dimethylformamide. In the determination of Cu^{II}, Ni and Co the use of NH_4ClO_4, HNO_3, $LiNO_3$ and oxalic acid eliminated inflection points due to replacement reactions (metal-to-edta ratio 2 : 1) but did not mask the chelating reaction (metal-to-edta ratio 1 : 1). Titrations of $MgCl_2$ and $SrCl_2$ could not be simplified. Nitric acid or $LiNO_3$ was used in the ratio 1 : 1 with the metal ion, and oxalic acid in the ratio 1 : 2. Differential titrations of mixed chlorides, masked and unmasked, gave satisfactory results. Water (<10%) did not affect the results.

1B13. **Coulometric determination of metal ions using copper - diethylenetriaminepenta-acetate complex as reactant.** Takata, Yoshinori; Arikawa, Yoshijiro; and Muto, Giichi (Res. Lab., Hitachi Ltd., Kuji-machi, Hitachi-shi, Ibaraki, Japan). *Bunseki Kagaku*, 1977, **26** (6), 407–411 (in Japanese).—A study was made of the use of Cu complexes instead of Hg complexes of edta and of diethylenetriaminepenta-acetate (dtpa) in the determination of metal ions by column chromatography with a flow coulometric detector (carbon cloth electrode). It was found that Cuedta was not suitable but that Cudtpa was more stable than the corresponding Hg complex; however, the detection limits with this reagent were poorer. The variation in current efficiency over 50 days was 2·2%. The effect of the temp. of the electrolytic cell was investigated and temp. >20° were found to be suitable. The electrolytic efficiencies for Cu^{2+}, Zn^{2+}, Pb^{2+}, Co^{2+} and Cd^{2+} separated by ion-exchange chromatography with Cudtpa were ≃100%.

1B14. **Analysis of metals by solid - liquid separation after liquid - liquid extraction using naphthalene.** Satake, Masatade; and Takagi, Y. (Fac. Eng., Fukui Univ., Fukui-shi, Japan). *Bunseki Kagaku*, 1977, **26** (6), 386–392 (in Japanese).—Water-insoluble complexes of various metals are quantitatively extracted into molten naphthalene. After cooling, the solid mixture is separated, dried on filter-paper and dissolved in dimethylformamide. The metal complexes in this soln. are determined spectrophotometrically. Suitable conditions (together with coeff. of variation) are given for the determination of Cu, Bi and Ni complexed with diethyldithiocarbamate; Cu, Bi and Co with ammonium pyrrolidine-1-carbodithioate; Cu, Zn and

Ni with 8-hydroxyquinaldine; Cu with 5,7-dichloro- and 5,7-dibromo-8-hydroxyquinoline; Cu with benzoin α-oxime; Fe^{III} and V^{V} with *N*-phenylbenzohydroxamic acid; and Ni with salicylaldoxime. Copper, Zn, Mg, Cd and Co were also determined with 8-hydroxyquinoline as complexing agent and measurement by a.a.s.

1B15. **Fluorescence properties of complexes of [Periodic-]group II and III metals with 5-sulpho-8-quinolinol [8-hydroxyquinoline-5-sulphonic acid] and their use in analytical chemistry.** Nishikawa, Yasuharu; Hiraki, Keizo; Morishige, Kiyotoshi; and Katagi, Teruhisa (Dept. Chem., Fac. Sci. and Technol., Kinki Univ., Higashi, Osaka, Japan). *Bunseki Kagaku*, 1977, **26** (6), 365–370 (in Japanese).—The water-soluble chelates of the named reagent (**I**) with Mg, Zn, Cd, Al, Ga and In have been used for the fluorimetric determination of these metals. To the sample soln., containing 0·1 to 20 μg of metal ion, were added 1 ml of 0·045% **I** soln. and 2 ml of 20% NH_4Cl or ammonium acetate soln. The soln. was made up to ≃20 ml, the pH was adjusted to the optimum (*i.e.*, for Mg 10, Zn 7, Cd 7·5, Al 4·5, Ga 2·5 and In 6·5 to 7·0) and the soln. was made up to 25 ml. The fluorescence intensity was measured against a soln. of fluorescein (0·125 μg ml^{-1}) as standard. In the determination of 0·1 to 4 μg of Al and Mg and 0·5 to 20 μg of Zn, Cd, Ga and In, the error was within ±2%. From a study of the fluorescence lifetimes of the chelates at different pH values it is concluded that they could be used in differential kinetic analyses.

1B16. **Gas-chromatographic separation and determination of some cations as diethyl phosphinodithioate chelates.** Kleinmann, Adolf; and Neeb, Rolf (Inst. Inorg. and Anal. Chem., Johannes Gutenberg Univ., Mainz, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **285** (2), 107–109 (in German).—Thermogravimetric data are presented for the bis-(diethyl phosphinodithioate) chelates of Zn, Cd, Co, Ni and Pb. Retention times are given for g.l.c. on Apiezon L, GE SE-30, OV-101, OV-17 and OV-25 columns, showing the possibility of separating all five metals. The calibration graph for the determination of the Cd chelate on OV-17 with flame ionisation detection is rectilinear from 10 ng (detection limit) to 10 μg. D. B. A.

1B17. **Thin-layer chromatography of metal ions on stannic arsenate: quantitative separation of, *e.g.*, mercury(II) from cadmium(II), zinc(II) and copper(II).** Qureshi, M.; Varshney, K. G.; and Fatima, N. (Chem. Sect., Z. H. Coll. Eng. and Technol., Aligarh Muslim Univ., India). *Sep. Sci.*, 1977, **12** (3), 321–328.—The lavers were prepared by mixing 0·05M-$SnCl_4$ and 0·05M-Na_2HAsO_4 in a ratio of 3:2, allowing the ppt. formed to settle for 24 h, then, after washing it with H_2O and treating with 2M-HNO_3, making the product (50 g) into a slurry with H_2O (100 ml) and spreading it on glass plates. The developing solvents used were 0·5M-HNO_3, M-HNO_3, 0·5M-HCl, M-HCl, M-HNO_3 - M-$NaNO_3$ (1:1), 0·1M-H_3AsO_4, and 10% tartaric acid soln. Quantitative separation of Hg (60 μg) from Cd (22·4 μg), Zn (56 μg) and Cu (56 μg) was thereby achieved, as well as separation of, *e.g.*, Cd from Sb, V from Nb, V from Ta, Ni from Fe, Pd from Nb, Hg from Pb, and Pb from Mo. The effect of heating the $Sn_3(AsO_4)_4$ gel under reflux in the mother liquor on R_F values is discussed. M. J. Sl.

1B18. **Metallurgical applications of energy-dispersive micro-probe analysis.** Dudek, H. J.; and Ziegler, G. (Inst. Werkstoff-Forschung, Koeln, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 243–260 (in German).—The technique was used in (*i*) the determination of Mg, Cu and Zn in aluminium alloys (the error being ≃5% for contents >10%); (*ii*) the determination of 90 to 100% of Al in alloys (the results being ≃1% high); and (*iii*) the examination of fracture surfaces of Ti - Al - V alloys, wherein phases can be identified and their structures determined by semi-quantitative analysis. Depth and lateral resolution are discussed. J. W. P.

1B19. **Determination of certain elements using a source of californium-252.** Agripov, G.; Askarov, A. I.; Tazhiev, N.; Sattarov, M. G.; and Shakirov, Sh. Yu. (Inst. Yad. Fiz., Tashkent, USSR). *Izv. Akad. Nauk Uzb. SSR, Ser. Fiz.-Mat. Nauk*, 1976, (5), 59–61 (in Russian).—Aluminium and Si were determined in ores by measuring the γ-ray activity of ^{28}Al produced in the reactions $^{27}Al(n,\gamma)^{28}Al$ and $^{28}Si(n,p)^{28}Al$. The sample (5 g) and standards were irradiated for 5 min with neutrons from a ^{252}Cf source (for the determination of Si, samples and standards were packed into a cadmium container to absorb thermal neutrons and were irradiated at 2 cm from the source), and, after a 1-min decay period, the γ-ray activities were measured with use of a NaI(Tl) detector. The sensitivity of the method was 0·01% for Al and 0·5% for Si, and the relative errors were 13 and 18%, respectively; 70 determinations could be effected in one working day. Sodium could be determined similarly, with a sensitivity of 0·1 mg, but an irradiation time of 72 h was required. Chem. Abstr.

1B20. **Qualitative analysis of a mixture of anions by partition chromatography on circular paper.** Boiko, V. F.; and Vasilevskaya, G. S. *Khim. Khim. Tekhnol. (Minsk)*, 1976, **10**, 94–97 (in Russian).—Several anions were separated by development with ethanol - H_2O - 1·5N-NH_3 (6:2:1), the two duplicate chromatograms produced being cut into sections for detection of bands with use of 10 reagents. R_F values are reported for NO_3^-, NO_2^-, $S_2O_3^{2-}$, CrO_4^{2-}, Br^-, S^{2-}, SO_4^{2-}, SO_3^{2-}, PO_4^{3-}, $B_4O_7^{2-}$, F^- and I^-. Chem. Abstr.

1B21. **Atomic-absorption determination, in metal sulphide concentrates, of elements that form gaseous hydrides.** Robert, R. V. D. (Natl. Inst. Metall., Milner Park, Johannesburg, S. Africa). *Natl. Inst. Metall., Repub. S. Afr., Rep.*, No. 1838, 1976. Pp. 37.—Interference by 30 elements (in 1-mg amounts), commonly present in sulphide concentrates, in the a.a.s. of As, Sb, Bi, Ge, Se and Te (0·5-μg amounts) as their hydrides has been studied. The hydrides were generated by treating soln. of the elements, in 10% (v/v) HCl, with 5% $NaBH_4$ soln., and measurements were made with use of N to carry the hydrides into the N - H - entrained air flame and of a background corrector. The findings (tabulated) were similar to those of Smith [*Analyst (London)*, 1975, **100**, 300], although in many instances the interference encountered was less marked; the most serious effects were caused by the precious metals and by Cu and Ni, and compensation for most interference proved possible. Germanium could not be determined in μg amounts by this technique, nor could all the elements be determined in one sample by co-pptn. as hydroxides with La as carrier. Detailed procedures are described; one portion of the concentrate is fused with Na_2O_2, the melt is leached

with H_2O, and the Cu and Ni are removed by filtration before determining As and Se. A second portion is fused with Na_2O_2, the melt is leached with HCl, and the Bi, Te and Sb are co-pptd. with $La(OH)_3$, leaving the Cu and Ni in soln. The ppt. is dissolved in HCl for analysis. Sensitivities (ng for 1% absorbance) are—As and Se 10, Bi 12, Sb 17, Te 19 and Ge 190. R. L. J.

1B22. **Research importance and possibilities of the analysis of high-purity metals.** Toelg, Guenther (Max-Planck Inst. Metallforsch., Schwaebisch Gmuend, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 1–26 (in German).—The effect of trace impurities on the properties of niobium and other metals such as tungsten, molybdenum and tantalum is discussed, and methods for separation and determination of N, B and P in μg and ng amounts in niobium are examined. Nitrogen is best determined by Kjeldahl distillation followed by coulometric titration, B by dissolution of the sample in HF and distillation of BF_3 from a PTFE vessel, followed by plasma-emission spectrometry, and P by pptn. of niobium phosphate and reduction to P by heating the ppt. with C at >1200°, followed by emission spectrometry. Also, an apparatus is described for the separation of pg amounts of Bi, Fe, Co and Zn by electrolysis. J. W. P.

1B23. **Effect of carriers in spectrographic determination of trace impurities by the dry-residue method.** Karyakin, A. V.; Pavlenko, L. I.; and Babicheva, G. G. (V. I. Vernadskii Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (6), 1071–1076 (in Russian).—The effects of 15 carriers (including various salts of Na and the nitrates of K, Ba, In and Pb) on the intensities of the analytical lines of Mn, Mg, Cr, Fe, Ni, Al, B and Si were studied. The flat top of a carbon electrode was moistened with 2 drops of 0·5% polystyrene soln. in benzene, dried, treated with the carrier soln. (to contain ≃2% of the carrier relative to the mass of dry residue from the test soln.), again dried and treated with 1 drop of the test soln. After drying the test soln., the residue was excited in a 10-A d.c. arc. Sodium silicate and sodium phosphate increased the intensities for Mn, Cr, Al and Fe; $Ba(NO_3)_2$ and NaCl were good carriers for Mn, Fe and Al. Lower limits of determination of the named elements in prepared soln. were 0·1 to 1 p.p.m. with use of the recommended carriers. M. P.

1B24. **Glyoxal bis-(2-hydroxyanil) as indicator for complexometric titrations of cobalt(II), nickel(II), manganese(II) and silver(I).** Osman, M. M. (Chem. Dept., Univ. Libya, Tripoli). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 43.—Cobalt or Ni (1 to 18 mg) is treated with an excess of standard edta soln. in neutral or slightly acid soln., and, after addition of 5 ml of M-NaOH and 1 to 2 ml of a 0·01M soln. of the indicator in ethanol and dilution to ≃100 ml, the unconsumed edta is titrated with standard $CaCl_2$ soln. to a colour change from yellow to orange. Manganese (2 to 33 mg) in neutral soln., after addition of excess of edta soln., is treated with ≃0·1 g of phenylhydrazine hydrochloride followed by 2 ml of M-NaOH and 1 to 2 ml of the indicator soln., the soln. is diluted to ≃50 ml and unconsumed edta is titrated with $CaCl_2$ soln. at pH 11·5. Silver (10 to 65 mg) is caused to react at pH 10 with 0·15 g of $K_2Ni(CN)_4$, and the liberated Ni^{II} is determined as described above. H. L. L.

Elements (Periodic-Group order)

Noble gases

He, Ne, Ar, Kr, Xe, Rn

1B25. **Gas-chromatographic determination of traces of hydrogen in helium.** Mirzayanov, V. S. *Zavod. Lab.*, 1977, **43** (6), 664–666 (in Russian).—Pass the sample (at 100 ml min^{-1}) through a liquid-N-cooled trap containing activated carbon to remove CH_4, and then through a reactor (40 cm × 0·6 cm) packed with a Pd - Al_2O_3 catalyst (prep. described) previously saturated with CO_2 at 200°. The H is converted into CH_4, which is determined with a flame ionisation detector (either continuously or intermittently after concentration in a cold trap). The method can be used to determine H in the range 5 to 500 p.p.b. A. S. W.

Group I

H, Li, Na, K, Rb, Cs,
Fr, Cu, Ag, Au

1B26. **Determination of the isotopic ratio of deuterium to protium at natural abundances of deuterium. II. Preparation of hydrogen for isotopic measurement.** Gottstein, Ottomar; and Hejl, Vaclav (Geol. Inst., Acad. Sci., Prague, Czechoslovakia). *Chem. Listy*, 1977, **71** (6), 649–653 (in Czech).—In a vacuum apparatus (shown diagrammatically), H is produced by reduction of water (25 to 40 μl) with specially prepared uranium at 650°. The liberated H is passed into a reaction tube at 80°, where it forms a hydride with more of the uranium. This hydride is decomposed at 600°, and ampoules are filled with the liberated H (at 53 to 82 kPa) and used for isotopic measurements by m.s. Preparation of the H from one sample takes ≃90 min. (For Part I see *Anal. Abstr.*, 1978, **34**, 1B234.) H. M. T.

1B27. **Method and apparatus for determination of boric acid in the coolant of light-water nuclear reactors.** Trifonov, A. I.; Stefanov, G. I.; Mikhailov, M. N.; Khristov, V. I.; and Guteva, E. S. (Inst. Nucl. Res. and Nucl. Energy, Sofia, Bulgaria). *Yad. Energ.*, 1976, (3), 42–48 (in Bulgarian).—The coolant water is passed through a steel cylinder (1·38 m × 0·5 m) containing a Pu - Be source of fast neutrons and a thermal-neutron counter (each in a stainless-steel tube in the centre of the cylinder); thermal neutrons obtained by moderation of the fast neutrons are absorbed by the H_3BO_3 in the coolant. With a source giving ≃5 × 10^6 n s^{-1} and the source and counter close to one another, the count rate varied exponentially with concn. of H_3BO_3, from 50,000 c.p.s. at zero concn. to 13,000 c.p.s. at 20 g l^{-1} of H_3BO_3. Variation in temp. is automatically compensated for by means of a thermistor. The error in determining 2 to 10 and 10 to 20 g l^{-1} of H_3BO_3 was <1·5 and 4%, respectively. C. D. K.

1B28. **Analytical applications of mixed-ligand complexes. II. Determination of hydrogen peroxide in the presence of peroxide derivatives.** Veber, M.; and Csanyi, L. J. (Dept. Inorg. and Anal. Chem., Attila Jozsef Univ., Szeged, Hungary). *Microchem. J.*, 1977, **22** (2), 238–244.—The method, which is applicable in the presence of H_2SO_5 and $H_2S_2O_8$, consists in the formation (in darkness during 40 min) of the Ti^{IV} - xylenol orange - H_2O_2 complex

at pH 1·25 for measurement at 520 nm against a similar soln. not containing H_2O_2. The concn. of Ti^{IV} and xylenol orange should each be ≃0·2mM. The calibration graph is rectilinear for 4 to 40μM-H_2O_2, and the standard deviation is ±0·2μM for 32 to 35μM-H_2O_2. (For Part I see *Anal. Abstr.*, 1977, **32**, 5B154.) W. J. B.

1B29. **Application of graphite-furnace atomic absorption to the determination of chromium, iron, manganese and nickel in sodium metal after distillation.** Huber, I.; Schreinlechner, I.; and Benischek, F. (Oesterreichische Studiengesellschaft fuer Atomenergie G.m.b.H., Forschungszentrum Seibersdorf, Austria). *At. Absorpt. Newsl.*, 1977, **16** (3), 64–66.—The sample (≃4 g) was weighed under Ar in a molybdenum or tantalum crucible, and, after distillation under high vacuum, the residue was dissolved in 3 ml of HCl and the vol. was made up to 20 ml with H_2O. Portions (100 μl) of the soln. were dried at 100° for 60 s, charred at 750° for 90 s and atomised at 2600° for 15 s, and the lines (nm) used for analysis were Cr 357·9, Fe 248·8, Mn 279·5, and Ni 232·0, all at 0·7-nm slit width and with Ar (1·5 l min^{-1}) as purge gas. Optimum working ranges were—Cr, 0·2 to 5 ng; Fe, 2 to 10 ng; Mn, 0·1 to 3 ng; and Ni, 4 to 20 ng, and detection limits ranged from 0·025 ng (1 p.p.b. in the sodium) for Mn to 0·3 ng (15 p.p.b.) for Ni. Recoveries from six samples of sodium (2·6 to 3·6 g) containing 10 μg of added Cr, Mn and Ni and 20 μg of added Fe averaged 96 to 99%. C. K. L.

1B30. **Co-precipitation of copper(II) with ferric hydroxide under conditions of complex formation.** Novikov, A. I.; Shaffert, A. A.; and Shchekoturova, E. K. (V. I. Lenin Tadzhik State Univ., Dushanbe, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1108–1115 (in Russian).—It is shown that ≃0·2 to 0·8mM-Cu^{II} can be concentrated from an aq. soln. (made M in KNO_3 and 4mM in Fe^{III}) by co-pptn. with $Fe(OH)_3$ at pH 7 to 12. The Cu^{II} can be separated from the Fe by dissolving the ppt. in acid and pouring this soln. into aq. 5M-NH_4NO_3 at pH >7·5. M. P.

1B31. **Spectrophotometric determination of copper(II) with chlorsulphophenol S.** Ishikura, Fumiko; and Tonosaki, Koichi (Fac. Sci., Hirosaki Univ., Japan). *Sci. Rep. Hirosaki Univ.*, 1976, **23** (1), 34–38 (in Japanese).—Bivalent Cu reacts with chlorsulphophenol S to form a blue complex having an absorption max. at 645 nm (ε ≃22,000). With a two-fold excess of reagent, the absorbance is constant over the pH range 2 to 4·3; Beer's law is obeyed for 3 to 50 μg of Cu^{II} in 25 ml at pH 4·0, and the Sandell sensitivity is 3 ng cm^{-2}. Aluminium, Bi^{III}, Ga, Th, V^{IV} and edta interfere seriously.

CHEM. ABSTR.

1B32. **Polarographic behaviour of copper ion in solution of o-carboxyphenylazoacetylacetone.** Elatrash, Ali M.; Elinany, Gaber A.; Ebeid, Fikry M.; Elhilaly, Ahmed E.; and El-Shatory, Beshir A. (Dept. Chem., Fac. Educ., Ain Shams Univ., Heliopolis, Cairo, Egypt). *J. Electroanal. Chem. Interfacial Electrochem.*, 1977, **79** (2), 375–379.—The polarographic behaviour of the complex formed between Cu^{2+} and this reagent (**I**) was studied, with use of a dropping-mercury electrode and a s.c.e., in soln. of 0·1M-Na_2SO_4 in aq. 25% dioxan and containing 0·01% of gelatin, 0·5mM-Cu^{2+} and mM-**I**. In unbuffered media of pH 2·9 to 10·5 and in 0·05M-acetate buffer of pH 4·4 to 6·5, the Cu complex gave one polarographic wave, the $E_{\frac{1}{4}}$ value of which shifted to more negative potentials with increasing pH. From pH 2·9 to 5·0, in unbuffered soln., the wave was irreversible and diffusion-controlled and corresponded to a two-electron reduction. In acetate buffer, the wave corresponded to a one-electron reduction, and was irreversible and largely diffusion controlled with an adsorptive component. At pH 6·0, there was a rectilinear relationship between the Cu concn. (0·125 to 0·909mM) and wave-height (at 0·5 V) of the complex. This complex was shown to have a Cu-to-**I** ratio of 1:1. J. P. A.

1B33. **Determination of copper in alloys by using electro-gravimetry combined with atomic-absorption spectrophotometry.** Nemodruk, A. A.; Egiazarova, N. V.; and Moreiskaya, L. V. (State Sci.-Res. and Design Inst. Alloys and Non-ferrous Metal Processing, Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 945–947 (in Russian).—Copper (≃58 to 93%) was determined in brasses and bronzes containing also ≃3 to 40% of Zn, 0·1 to 3% of Fe, 0·7 to 1·7% of Mn, 0·05 to 1·2% of Pb, 0·45 to 3·7% of Sn and <9% of Al. The samples (0·5 to 1 g) were dissolved in 30% HNO_3 (for brasses) or in 30% HNO_3 - HF (15:1) (for bronzes). The Cu (>99% of that present) was deposited on a platinum electrode by a conventional procedure, and the electrode was weighed; Cu remaining in the electrolyte was determined by a.a.s. (air - acetylene flame; measurement at 324·7 nm). M. P.

1B34. **Phosphorodithioic acids as reagents [for copper, silver and palladium] in electrochemical analysis.** Chebotarev, V. K.; Chigirintsev, V. D.; Frolova, V. I.; and Bagdasarov, K. N. *Izv. Sev.-Kavk. Nauch. Tsentr. Vyssh. Shkol. Estestv. Nauk*, 1976, (3), 46–48 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (17), Abstr. No. 17G84.

1B35. **Spectrophotometric and potentiometric studies on copper(II) and nickel(II) complexes with 5′-chloro-2′-hydroxy-4′-methylacetophenone oxime.** Gupta, S. P.; and Lal, Keemti (Chem. Lab., Dev Nagri Coll., Meerut, India). *Acta Cienc. Indica*, 1976, **2** (2), 119–122.—Bivalent Cu and Ni^{II} were determined spectrophotometrically by measuring the absorbance of their green 1:2 complexes (the compositions of which were deduced by, *inter alia*, potentiometric titration) with the oxime reagent at 640 and 580 nm, respectively, after extraction into $CHCl_3$ from aq. ethanol at pH 3·5 (Cu) and pH 8 (Ni). Beer's law was obeyed for up to 0·95 μg ml^{-1} of Cu and 0·44 μg ml^{-1} of Ni. Tervalent Fe interfered in the determination of Cu. CHEM. ABSTR.

1B36. **Determination of traces of copper, nickel and cobalt [in geological samples] by solvent extraction and atomic-absorption spectrophotometry.** Chowdhury, A. N.; De, D. K.; and Das, A. K. (Central Chem. Lab., Geol. Surv. India, Calcutta). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 41.—To ≃25 ml of soln. prepared from the sample are added 20 ml of 0·5M-ammonium citrate, aq. NH_3 to bring the pH to 9·5 to 10, and H_2O to 50 ml. This soln. is extracted with 5 ml of 2-nitroso-1-naphthol soln. in isobutyl methyl ketone, and the extract is aspirated into an air - acetylene flame. There is no interference from other elements. Copper or Ni can be determined in the presence of 20 times its wt. of the other two metals, and Co can be determined in the presence of 1000 times its wt. of Cu or Ni. H. L. L.

1B37. **Effect of ethylenediamine on extraction of copper with valeric or 2-bromovaleric acid.** Pyatnitskii, I. V.; Sukhan, V. V.; and Ishchenko, V. B. (T. G. Shevchenko Kiev State Univ., USSR). *Ukr. Khim. Zh.* (*Russ. Ed.*), 1977, **43** (4), 411–416 (in Russian).—Valeric acid (**I**) or 2-bromovaleric acid (**II**) in $CHCl_3$ soln. extracts Cu from aq. soln. with a max. degree of extraction of ≃95% at pH 6 (for **I**) or ≃90% at pH 4·5 (for **II**). In the presence of ethylenediamine, however, extraction of Cu by **I** becomes negligible, whereas that by **II** is enhanced. Reasons for the effect are discussed. G. S. S.

1B38. **Use of an energy-dispersive X-ray spectrometer for high-speed ultimate analysis of brass alloys.** Kis-Varga, M.; and Bacso, J. *Atomki Kozl.*, 1976, **18** (4), 577–583 (in Hungarian).—The system described, which is suitable for the quality-control analysis of samples of brass containing Cu (50 to 70%), Zn and Pb (0 to 4%), incorporates a Si(Li) detector, a multi-channel analyser and analogue data-processing electronics; measurements are controlled, and spectra are evaluated, by means of a small computer. The system is easy to operate and yields reliable results. HUNG. TECH. ABSTR.

1B39. **Determination of impurities in copper-based solder.** Koval'chuk, K. G.; Perminova, V. M.; and Ippolitov, V. M. *Zavod. Lab.*, 1977, **43** (6), 682–684 (in Russian).—A spectrographic method is described for determining down to ≃10 p.p.m. each of Zn, Bi, Pb, Mn, As, Sb and Cd in such samples. The solder (1 g) is dissolved in 10 to 15 ml of HNO_3 (1 : 1), Ge_2O_3 (≃0·1 g) is added as internal standard, the mixture is evaporated and the residue is heated for 30 min in a muffle-furnace at 350° to 400°. The cooled residue is ground and packed into a carbon electrode, and the spectrum is excited in a 6 to 7-A d.c. arc. Similarly prepared standards are treated analogously, and the concn. of each element is calculated from a calibration graph. The analytical wavelengths are listed. A. S. W.

1B40. **Determination of tin in copper-base alloys high in manganese.** Khanna, V. B.; Rai, B. L.; and Maheshwari, S. K. (Off. Director Insp., New Delhi, India). *Res. Ind.*, 1976, **21** (2), 111–112.—In the gravimetric determination of Sn in such alloys by pptg. $SnO(OH)_2$ with dil. HNO_3, the white ppt. was contaminated by particles of hydrated MnO_2. To avoid such interference, cane sugar was added to the sample soln. to reduce Mn^{IV} to Mn^{II}. The ppt. was filtered off, ignited at 900°, cooled and weighed. The results agreed with those obtained by a conventional titrimetric method. CHEM. ABSTR.

1B41. **Spectrographic determination of germanium and rhenium in copper - nickel ores, products of their processing and production wastes by means of fractional distillation.** Shokina, N. T.; and Shuvalova, M. E. (State Sci.-Res. and Design Inst. Rare Metals, Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1127–1130 (in Russian).—To determine 1 to 100 p.p.m. of Ge, 20 mg of sample is mixed with 20 mg of silica - sulphur (1 : 1) carrier containing 0·5% of Sb_2S_3 as internal standard. The mixture is placed in a 1-cm-deep cavity in a graphite anode and covered with a graphite stopper pierced by a hole (diam. 1 mm), and is then excited in a 7-A d.c. arc (with a graphite counter-electrode); the exposure time is 60 s. To determine 3 to 100 p.p.m. of Re, 20 mg of sample is mixed with 100 mg of lead dioxide containing 0·1% of $CdSO_4$ as the internal standard, and the mixture is excited as for Ge but in a 5-A arc and with an exposure time of 30 s. The coeff. of variation is <20% in each instance. One set of standards can be used in analysing many materials. The results obtained agreed with those of n.a.a. and spectrophotometric methods. M. P.

1B42. **Fluorimetric-reaction-rate method for determination of silver.** Wilson, Robert L.; and Ingle, James D., jun. (Dept. Chem., State Univ., Corvallis, Ore. 97331, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1066–1070.—The reaction between 8-hydroxyquinoline-5-sulphonic acid and $S_2O_8^{2-}$ is catalysed by Ag^+; this fact is used as the basis of a method for determining Ag, by means of the instrument described (*Anal. Abstr.*, 1978, **34**, 1J77), in the range 6 p.p.b. to 30 p.p.m. (with coeff. of variation of <0·5%). A dithizone extraction procedure is recommended to avoid interference from V^V, Sn^{II}, Sn^{IV}, Al, Zr, Hf, Pt^{II}, Au^{III}, NO_3^- and Cl^-. There is good agreement in the results obtained for the Ag content (0·8%) of a NBS spelter with those by conventional a.a.s. R. W.

1B43. **Atomic-absorption determination of silver after extraction into a chloroform solution of cinnamic acid containing pyridine.** Pyatnitskii, I. V.; and Pilipyuk, Ya. S. (T. G. Shevchenko Kiev State Univ., USSR). *Ukr. Khim. Zh.* (*Russ. Ed.*), 1977, **43** (6), 639–641 (in Russian).—The test soln. (1 ml) containing 0·1 mg of Ag is shaken with 10 ml of $CHCl_3$ that is 0·2M in both cinnamic acid and pyridine. The pH of the mixture is adjusted to 6·5 to 11 with alkali, and, after further shaking, the phases are separated, and the $CHCl_3$ phase is diluted to 25 ml with methanol. This soln. is submitted to a.a.s. (air - propane - butane flame) for Ag, with measurement at 328·1 nm. The graph of absorption *vs.* Ag content is rectilinear, and the sensitivity is better by one order of magnitude than that attained by atomisation of aq. soln. A. B.-S.

1B44. **Spectrophotometric determination of silver(I) with 1,3-diamino-8-methoxyphenothiazine.** Soni, K. P.; Mathur, S. P.; and Chaudhary, Ramesh C. (Res. Lab., Gov. Coll., Churu, India). *Ann. Soc. Sci. Bruxelles, Ser.* 1, 1976, **90** (4), 337–338.—The absorbance of the pink 1 : 1 complex formed at pH 4·6 to 9·4 between Ag and the named reagent is measured at 480 nm ($\epsilon \simeq 14{,}600$). The complex is insoluble in H_2O but soluble in alcohol or CCl_4. Beer's law is obeyed for up to 1·8 p.p.m. of Ag, and the Sandell sensitivity is 0·143 $\mu g\ cm^{-2}$. Bivalent Pb, Sn^{II}, Cu^{II}, Fe^{III}, Pd^{IV} and Pt^{IV} are pptd. by the reagent at pH 6·2, and can be removed by centrifugation. Oxalate, F^- and edta mask the reaction. CHEM. ABSTR.

1B45. **Reaction between silver and ferrocene in aqueous organic media.** Solomatin, V. T.; Ignatov, V. I.; and Rzhavichev, S. P. (Central Sci.-Res. Inst. Ferrous Metall., Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 948–953 (in Russian).—It is shown that Ag can be titrated amperometrically with ethanolic 0·01M-ferrocene in media of H_2O - acetic acid, H_2O - acetone or H_2O - ethanol (2 : 1 to 3 : 1) that are M to 2M in H_2SO_4 or 2 to 4M in $HClO_4$; the oxidation current of ferrocene at a Carbositall electrode (operated *vs.* an alkaline permanganate electrode) is measured, and the lower limit of determination is 0·12 $\mu g\ ml^{-1}$ of Ag. Zinc, Ni and Mg do not interfere, but Cu^{II}, Hg^{II}, Hg^{I} and Fe^{III} do. The method was used for determining ≃10% of Ag in a Mg - Al alloy (catalyst); the coeff of variation was 1% (8 results). M. P.

1B46. **Atomic-absorption determination of silver in rocks.** Pchelintseva, N. F. (M. V. Lomonosov Moscow State Univ., USSR). *Zavod. Lab.*, 1977, **43** (6), 693 (in Russian).—The sample, mixed with an equal wt. of graphite powder, is placed in a graphite chamber attached to the spectrometer in place of the normal burner. The Ag is selectively vaporised by heating the chamber to 1500° by an electric current (≃105 A) from a transformer. The absorption during vaporisation and atomisation of the Ag is measured, freedom from non-resonance absorption being confirmed in a supplementary test for the Cd line at 326·1 nm. The Ag concn. is calculated from a calibration graph established from standards based on graphite or quartz. The detection limit is ≃1 p.p.b. of Ag, and results are independent of the rate of vaporisation or the composition of the sample. A. S. W.

1B47. **Selective detection of gold.** Rao, B. V. (Defence Metall. Res. Lab., Hyderabad, India). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 44.—As little as 50 μg of Au can be detected by the blue colour of the colloidal Au produced, in a soln. mM in $AuCl_3$ and containing pyrophosphate, by reduction with $Ce_2(SO_4)_3$ at pH 7 to 8. Silver, Pd^{II}, Rh, Ir^{III}, Pt^{IV}, Pb, Hg, Zn, Cd, Ca, Co, Cu, Al, Fe^{III}, Cr^{III}, Bi, Sb, Sn^{IV}, W^{VI} and Mo^{VI} do not interfere if present in amounts less than that of the Au, but V^{V}, Mn^{II}, Br^- and I^- interfere at all levels. Quantitative application of the method is suggested. H. L. L.

1B48. **Spectrophotometric determination of gold(III) with aniline.** Rzeszutko, W.; and Kopec, T. (Dept. Inorg. Chem., Med. Acad., Krakow, Poland). *Fresenius' Z. Anal. Chem.*, 1977, **285** (2), 125.—A blue - violet Au^{III} - aniline complex is formed in alkaline media with an absorption max. at 560 nm ($\epsilon = 6200$), and may be extracted from aq. soln. into benzyl alcohol. Beer's law is obeyed in the range 5·7 to 28·5 μg ml^{-1}; Ag interferes, but Cu up to 25% of the Au content is tolerated. D. B. A.

1B49. **Atomic-absorption determination of gold by using a flame atomiser and a flameless graphite atomiser after solvent extraction [of gold] with petroleum sulphides.** Torgov, V. G.; and Khlebnikova, A. A. (Inst. Inorg. Chem., Acad. Sci. USSR, Siberian Branch, Novosibirsk). *Zh. Anal. Khim.*, 1977, **32** (5), 960–964 (in Russian).—Traces of Au in standard rocks, hot-spring water and plants were determined by ashing the sample (10 g) at 600°, dissolving the ash in aqua regia, evaporating the soln. to dryness and dissolving the residue in 2M-HCl (100 ml). The Au was extracted from this soln. with a 0·1M soln. of petroleum sulphides (boiling-range 212° to 265°) in toluene (1 to 5 ml). The extract was either aspirated into an air - toluene flame (for 0·01 to 10 p.p.m. of Au) or atomised in a graphite furnace (for 0·1 to 8 p.p.b. of Au). The coeff. of variation was <13% (6 results) when using the flame method and <16% (6 results) for the flameless procedure. M. P.

1B50. **Atomic-absorption determination of traces of gold in geochemical samples by using pulsed atomisation.** Men'shikov, V. I.; Khlebnikova, A. A.; Tsykhanskii, V. D.; and Malykh, V. D. (Inst. Geochem., Acad. Sci. USSR, Siberian Branch, Irkutsk). *Zh. Anal. Khim.*, 1977, **32** (5), 954–959 (in Russian).—Traces of Au were determined in rocks, minerals, hot-spring water and plants. The samples were decomposed by conventional procedures, and Au was extracted from a medium 2M in HCl into 0·2M-dibutyl sulphide in toluene. The extract was analysed for Au by using an a.a.s. instrument equipped with a graphite furnace or with a combined graphite furnace - flame atomiser. The extract (0·05 to 0·8 ml) was dried at 100° for 30 s, heated at 200° for 30 s and atomised at 2400° (10-s pulse); measurements were made at 242·8 nm. The calibration graph was rectilinear for 0·5 to 20 ng ml^{-1} of Au. The coeff. of variation was 5% (25 results) for 5 to 10 ng ml^{-1} of Au in prepared soln., and <30% when determining 0·01 to 0·1 p.p.b. of Au in geological samples. Results agreed with those of n.a.a. and spectrography. M. P.

1B51. **Determination of gold, platinum and palladium in silver - gold alloys by spectroscopy.** Khrapai, V. P.; Zastrozhin, S. I.; and Arnautov, N. V. *Tr. Inst. Geol. Geofiz. Akad. Nauk SSSR, Sib. Otd.*, (315), 17–19 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G201.

Group II

Be, Mg, Ca, Sr, Ba, Ra, Zn, Cd, Hg

1B52. **Spectrophotometric micro-determination of alkaline-earth and lanthanoid elements with hydroxynaphthol blue [3-hydroxy-4-(2-hydroxy-4-sulpho-1-naphthylazo)naphthalene-2,7-disulphonic acid] and ethylenediaminetetra-acetic acid.** Brittain, Harry G. (Dept. Chem., Ferrum Coll., Ferrum, Va. 24088, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 969–972.—Absorption spectra (400 to 700 nm) are reproduced for the named reagent (**I**) (protonated and unprotonated) and for its complex with Sr. **I** can be used in conjunction with edta for the spectrophotometric determination of the elements cited, the lower detection limits ranging from 1·6 μg ml^{-1} for Sm^{III} to 19·5 μg ml^{-1} for Dy^{III}. In the suggested procedure, **I** and edta are used as mM and 0·01M soln., respectively, in aq. phosphate or acetate buffer of pH 6. R. W.

1B53. **Gas-chromatographic analysis of metals [*e.g.*, beryllium, aluminium and chromium] as volatile complexes.** Sokolov, D. N. *Usp. Khim.*, 1977, **46** (4), 740–749 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G97.

1B54. **Column partition chromatography—an effective method for separation of elements.** Dubrovina, L. T.; Luginin, V. A.; and Tserkovnitskaya, I. A. *Probl. Sovrem. Anal. Khim.*, 1976, **1**, 169–174 (in Russian).—Advantages of reversed-phase column partition chromatography over conventional extraction techniques are discussed, and a method has been developed for the separation of ≃0·05 to 0·1% of Be from Al by the chromatographic technique. An acidic soln. containing Al and Be was treated with a three- to five-fold excess (with respect to Al) of Na_2-edta, and the soln. was adjusted to pH ≃6 with aq. NH_3 (1 : 1); ≃0·1 to 0·2 ml of the alkaline soln. was applied to a column that had been treated with 30 vol. of bis-(2-ethylhexyl) phosphate soln. in hexane and equilibrated with 5% NaCl soln. at pH ≃6. The unconsumed edta was eluted with the NaCl soln., and Be was eluted with 5·5M-HCl. CHEM. ABSTR.

1B55. **Determination of magnesium and iron with use of 8-hydroxyquinoline and coulometric titration with electro-generated bromine.** Chazova, L. A.;

Lopatin, B. A.; and Rubanova, E. V. *Izv. Sev.-Kavk. Nauch. Tsentr. Vyssh. Shkol. Estestv. Nauk*, 1976, (3), 51–54 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (17), Abstr. No. 17G90.

1B56. **Comparison of metallochromic indicators for the determination of calcium.** Belen'kii, S. M.; Dul'neva, T. N.; Klyachko, Yu. A.; Petrova, G. S.; and Vysokova, N. N. *Zavod. Lab.*, 1977, **43** (6), 649–650 (in Russian).—Murexide, calcichrome and Cal-Red [3-hydroxy-4-(3-hydroxy-4-sulpho-1-naphthylazo)-2-naphthoic acid] have been compared as indicators for the determination of Ca (up to 700 mg l^{-1}) in aq. soln.; Cal-Red gives the smallest error and the sharpest end-point. A. S. W.

1B57. **Quantitative X-ray diffraction analysis of multi-component systems containing calcium phosphates.** Churakova, E. K.; Poloznikov, A. I.; and Aziev, R. G. (M. V. Lomonosov Moscow State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 1031–1034 (in Russian).—This method has been used for determining ≃8 to 74% of $Ca_3(PO_4)_2$, 17 to 50% of β-$Ca_2P_2O_7$, 2 to 50% of β-$Ca(PO_3)_2$ and 3 to 29% of CaO in the products of the reduction of calcium phosphate. Powdered samples were mixed with starch, and with KCl (to contain 3% of KCl) as the internal standard, and the mixtures were analysed with use of a diffractometer equipped with a copper anode (the Cu $K\alpha$ line was used). The concn. of the individual components were calculated from the diffraction-peak intensities. The relative error was $<\pm 5\%$. M. P.

1B58. **Determination of various forms of phosphates in binary mixtures by X-ray phase analysis.** Churakova, E. K.; Poloznikov, A. I.; and Aziev, R. G. (Fac. Chem. Technol., Univ. Moscow, USSR). *Vestn. Mosk. Univ., Khim.*, 1977, **18** (2), 233–234 (in Russian).—See preceding abstract.

1B59. **Determination of barium in calcium carbonate rocks by carbon-furnace atomic-emission spectrometry.** Hutton, R. C.; Ottaway, J. M.; Rains, T. C.; and Epstein, M. S. (Dept. Pure and Appl. Chem., Univ. Strathclyde, Glasgow, Scotland). *Analyst* (*London*), 1977, **102** (1215), 429–435.—Dissolve 1 g of sample in 10 ml of 4% (v/v) HNO_3, filter if necessary, add 1 ml of a soln. of Cs [1 mg ml^{-1} in 1% (v/v) HNO_3] as ionisation suppressor and dilute to 100 ml with H_2O. Apply 50-μl aliquots to the graphite atomiser of an atomic-absorption spectrophotometer operating in the emission mode, dry at 120°, atomise at 2800° and measure the emission at 553·5 nm. Spectral interference from CaOH bands is much less than for flame atomisation, and is eliminated by background correction; down to 0·036 p.p.m. of Ba in rock can be detected. R. W.

1B60. **Radiochemical determination of strontium-89 and strontium-90: comparison of methods based on error analysis.** Bowman, W. B., II; Swindle, D. L.; and Henderson, D. L. (Carolina Power & Light Co., Raleigh, N.C. 27602, U.S.A.). *Health Phys.*, 1976, **31** (6), 495–500.—A method requiring two separation steps, *viz*, the separation of Sr (after removal of ^{90}Y), measurement of its activity and, after build-up of ^{90}Y, separation and measurement of this isotope, is compared with a method requiring only initial separation of the Sr fraction, then measurement of the Sr activities immediately and again after 3 to 13 days. Results show that the former method gives a constant, acceptable relative error for ^{90}Sr, while the error for ^{89}Sr ranges varies from ≃50% for low ^{89}Sr activity to <10% at high ^{89}Sr activity. The only situation that favours use of the single-separation technique is a ratio of ^{89}Sr to ^{90}Sr between 0·1 and 0·3. A. B. K.-S.

1B61. **Separation of strontium ion from a large amount of calcium ion by use of a macrocyclic ether.** Kimura, Toshimasa; Iwashima, Kiyoshi; Ishimori, Tatsujiro; and Hamaguchi, Hiroshi (Japan Chem. Anal. Centre, Funato, Itabashi-ku, Tokyo). *Chem. Lett.*, 1977, (5), 563–564.—Separation of 0·7 to 20 mg of Sr from 1·5 g of Ca is effected by extraction of the Sr from an aq. medium 20mM in Na picrate, 55mM in acetic acid and 55mM in Na acetate into 11mM-dicyclohexyl-18-crown-6 {eicosahydrodibenzo[*b*,*k*]-[1,4,7,10,13,16]hexaoxacyclo-octadecin} in $CHCl_3$; picrate acts as counter-ion. More than 98% of the Sr is extracted in a single step, and the small amount of Ca^{2+} co-extracted can be removed by washing the extract with 85mM-acetic acid - 85mM-Na acetate - 15mM-Na picrate. The recovery of Sr is then 97%, and the decontamination factor for Ca is 10^4 to 10^5. The Sr can be re-extracted into 2M-NH_4Cl - 0·2M-HCl. The procedure should be applicable in determining Sr radionuclides in environmental samples rich in Ca. L. M. C.

1B62. **Separation and gravimetric determination of barium using sulphadimethoxine salicylaldimine.** Jain, Prabuddha; and Chaturvedi, Kamal K. (Dept. Chem., Holkar Sci. Coll., Indore, India). *Microchem. J.*, 1977, **22** (1), 92–95.—A soln. of this reagent [*N*-(2,6-dimethoxypyrimidin-4-yl)-4-salicylideneaminobenzenesulphonamide] was added, in slight excess, to the hot test soln., and the pH was adjusted to 6. After heating the mixture for 30 min on a water bath, the white ppt. formed was filtered off and washed with hot water, and then with ethanol - water, dried at 115° to 120° and weighed as $(C_{19}H_{17}O_5N_4S)_2Ba$. The effects of 19 cations (together with suitable masking agents) are given, and the nature of the complex is discussed. T. G. A.

1B63. **Calibration studies for quantitative X-ray photo-electron spectroscopy of ions.** Bancroft, G. Michael; Brown, James R.; and Fyfe, W. S. (Dept. Chem. and Geol., Univ. Western Ontario, London, Ont., Canada). *Anal. Chem.*, 1977, **49** (7), 1044–1048.—Cleaved calcite (Iceland spar) surfaces carrying sub-monolayer amounts of Ba and Pb, applied by lightly etching the surface with emery cloth and evaporating on to it dil. aq. soln. of $Ba(ClO_4)_2$, $BaCl_2$ or $Pb(NO_3)_2$, were examined by the technique cited. Down to ≃1 ng cm^{-2} of Ba^{2+} or Pb^{2+} could be detected, and reproducible calibration graphs were obtained by use of ratios of Ba^{2+} or Pb^{2+} to the Ca in the matrix. R. W.

1B64. **Anodic amalgam voltammetry. XII. Inverse-voltammetric determination of zinc in basal electrolytes containing triethanolamine.** Neeb, Rolf; and Kiehnast, Ingrid (Inst. Anorg. u. Anal. Chem., Johannes Gutenberg Univ., Mainz, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **285** (2), 121–123 (in German).—Zinc can be determined in the range 0·1μM to 0·1mM by inverse voltammetry in alkaline electrolytes containing triethanolamine (**I**), *e.g.*, M-**I** - M-NH_3. The presence of **I** greatly reduces interference from readily pptd. ions, *e.g.*, Al^{3+}, Mg^{2+}, Cr^{3+} and PO_4^{3-}. Optimum conditions for the determination of Zn are derived from the reported

experimental data. Determinations in the presence of Cd, Pb, Cu and Fe^{3+} are also discussed. (For Part XI see *Anal. Abstr.*, 1973, **24**, 692.) D. B. A.

1B65. **Sub-stoicheiometric determination of zinc by isotope-dilution method using a chelating agent-loaded resin.** Akaiwa, Hideo; Kawamoto, Hiroshi; and Ogura, Kazuko (Dept. Chem., Fac. Technol., Gunma Univ., Kiryu, Japan). *Talanta*, 1977, **24** (6), 394–395.—Zinc is separated from other cations on an anion exchanger loaded with a sub-stoicheiometric amount of 8-hydroxyquinoline-5-sulphonic acid (*cf.* Akaiwa *et al.*, *Anal. Abstr.*, 1976, **30**, 5H18). *Procedure*—To a test soln. add 600 μg of Zn^{II} containing ^{65}Zn as a tracer and acetate buffer (pH 4·75), and adjust the vol. to 100 ml. Add 100 mg of anion exchanger, *e.g.*, Diaion SA 100 (Cl^- form), stir for 2 h, filter, and measure the activity of the resin phase. Treat a standard soln. in the same way, and measure the activity of the resin phase. Calculate the amount of Zn present in the test soln. from the ratio of the activity to that of the standard soln. Interfering metals can be removed beforehand by a dithizone extraction procedure. The method was applied to water analysis. T. J. S.

1B66. **Complex formation of metals of the zinc sub-group with 4-methyl-2-(2-hydroxynaphthylazo)-thiazoie and 4-adamantyl-2-(2-hydroxynaphthylazo)-thiazole [1-(4-methyl)- and 1-(4-adamantyl-2-thiazolylazo)-2-naphthol, respectively].** Pilipenko, A. T.; and Karetnikova, E. A. (T. G. Shevchenko Kiev State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1116–1150 (in Russian).—The named compounds (**I** and **II**, respectively) form red - violet water-insoluble 1:2 (metal-to-ligand) complexes with Zn, Cd and Hg^{II}. The complexes with **I** can be extracted into $CHCl_3$ from media of pH 8·5 to 11 for Zn, 8 to 10 for Cd and 5 to 7·5 for Hg, and exhibit max. absorption at $\simeq$580 to 594 nm (ϵ = 74,500 to 81,000); Beer's law is obeyed for 0·07 to 1·82 μg ml^{-1} of Zn, 0·11 to 2·67 μg ml^{-1} of Cd and 0·2 to 4·8 μg ml^{-1} of Hg. The complexes with **II** are extracted into $CHCl_3$ at pH 12 to 13 for Zn, 9·5 to 11 for Cd and 6·5 to 8 for Hg and exhibit max. absorption at 555, 595 and 590 nm, respectively (ϵ = 48,600 to 56,000); Beer's law is obeyed for 0·13 to 2·6 μg ml^{-1} of Zn, 0·14 to 4·5 μg ml^{-1} of Cd and 0·25 to 8 μg ml^{-1} of Hg. Tervalent Tl, Fe^{II}, Co^{II}, Ni, Cu, Ag, Mn^{II} and Pb interfere when using **II**. An extraction - spectrophotometric method has been devised for determining 1 to 40 p.p.m. of Zn in gallium metal or salts with **II** as reagent; Cd could be masked with I^-. M. P.

1B67. **Spectrophotometric study of the ternary systems bivalent metal - 1,10-phenanthroline - 'phloxine'. II. With zinc, cobalt or nickel as the bivalent metal.** Popa, Grigore; and Dumitrescu, Nina. *Rev. Chim.* (*Bucharest*), 1977, **28** (5), 486–487 (in Romanian).—The ternary complexes $[Me(phen)_2]^{2+}[phlox]^{2-}$, where Me is Zn, Co or Ni, phen is 1,10-phenanthroline and phlox is 'phloxine' (tetrabromodichlorofluorescein, Na salt), in aq. soln. of pH 5 to 9, have an absorption max. at 570 nm and can be used for the determination of the metals, Beer's law being obeyed for 3·27 to 40 μg of Zn, 2·94 to 35·34 μg of Co or 2·93 to 35·22 μg of Ni in 25 ml of soln. The complexes can be extracted into $CHCl_3$. (For Part I see *Anal. Abstr.*, 1977, **33**, 6B37.) C. D. K.

1B68. **Use of trioctylamine for sub-stoicheiometric separation of indium [in analysis of, *e.g.*, zinc].** Yakovlev, Yu. V.; Kolotov, V. P.; and Dogadkin, N. N. (V. I. Vernadskii Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (6), 1244–1247 (in Russian).—To determine traces of In and Cd in zinc, the sample (0·3 g) was irradiated in a neutron flux of $\simeq 1{\cdot}2 \times 10^{13}$ n cm^{-2} s^{-1} for 20 h, and dissolved in HNO_3 after adding In^{III} carrier. The soln. was treated with conc. H_2SO_4 (1·7 ml), concentrated and diluted with H_2O to 8 ml, then treated with 2M-KI (2 ml), and the In^{III} was extracted with a sub-stoicheiometric amount of 0·1M-trioctylamine in $CHCl_3$. The extract was washed twice with an aq. soln. 0·2M in KI and 3M in H_2SO_4, and 50% of the In (relative to the initial amount of carrier added) was re-extracted by a sub-stoicheiometric vol. of a soln. of the Tl^{III} - I^- complex in 3M-H_2SO_4. The γ-ray activity of the aq. phase was measured with a Ge(Li) detector at the peak energies of ^{116m}In (to determine In) and ^{115m}In [to determine Cd via the $^{114}Cd(n,\gamma)^{115}Cd \rightarrow {}^{115m}In$ reaction sequence]. The coeff. of variation were <18% (4 to 7 results) when determining 0·0024 p.p.m. of In and 270 p.p.m. of Cd, and limits of determination were 0·4 p.p.b. for In and 0·4 p.p.m. for Cd. Zinc, Ga, Fe, Co, As, Sn, Cu, Hg, Cd and Ag did not interfere in the extraction of In. The method can be also used for determining Cd, In and Sn (which yields ^{113m}In) in iron, cobalt, nickel, gallium, zirconium or hafnium. M. P.

1B69. **Simultaneous [voltammetric] determination of thickness and composition of electrc-deposited coatings of cadmium - tin alloys [on a copper base].** Slepushkin, V. V.; Kuz'mina, N. N.; and Yartsev, M. G. (V. V. Kuibyshev Polytech. Inst., Kuibyshev, USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 965–970 (in Russian).—The method involves polarisation of the sample as an electrode in a supporting electrolyte of M-$NaClO_4$. The voltammograms show partly separated current peaks for the oxidation of Cd and Sn. The individual oxidation currents are rectilinearly related to the concn. of Cd or Sn (15 to 50%) in the coating, and the sum of the oxidation currents is rectilinearly related to the thickness of the coating (5 to 20 μm). The method is suitable only for freshly deposited coatings, as coatings several months old form intermetallic phases with the copper base, yielding more complex voltammograms. M. P.

1B70. **Liquid - liquid extraction of cadmium with Alamine 336 from aqueous chloride and bromide media.** McDonald, Curtis W.; and Pahlavan, Gholam Hussein (Dept. Chem., Texas Southern Univ., Houston, Tex. 77004, U.S.A.). *Sep. Sci.*, 1977, **12** (3), 271–279.—The effects of, *e.g.*, reagent concn., pH, mixing and settling times and vol. ratio on the extraction of Cd from such media are discussed. Under optimum conditions, Cd (4 p.p.m.) can be quantitatively extracted from M-HCl or -HBr at room temp. with an equal vol. of 5% Alamine 336 soln. in xylene within 15 s. The ratio of organic to aq. phase can be increased to 20:1 without loss of extraction efficiency; >98% of the Cd can be stripped from the organic phase with 0·1M-edta, M-NaOH or aq. 2·5M-NH_3. The method can be applied to the determination of Cd in waste waters. M. J. Sl.

1B71. **Kinetic determination of mercury, lead or cadmium.** Dolmanova, I. F.; Zolotova, G. A.; and Mazko, T. N. (M. V. Lomonosov Moscow State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 1025–1027 (in Russian).—The method involves reaction between the metal being determined and a catalyst - inhibitor complex; the metal binds the inhibitor, and

the catalyst is liberated, the rate of liberation being proportional to the concn. of the metal. It is shown that Hg^{II}, Pb^{II} or Cd^{II} can be determined by using the catalytic action of Fe^{III} on the oxidation of *p*-phenetidine with KIO_4 in the presence of 2,2′-bipyridyl; thiourea is used as inhibitor, and the reaction rate is determined spectrophotometrically. Under optimum conditions, *i.e.*, pH 5·7, 6·67 ng ml^{-1} of Fe^{III}, 1·7μM-thiourea, 0·1mM-*p*-phenetidine, 1·27mM-2,2′-bipyridyl and 0·35mM-KIO_4, the reaction rate is rectilinearly related to concn. (ng ml^{-1}) in the range 10 to 150 for Hg, 30 to 200 for Pb and 60 to 300 for Cd. In these ranges, the coeff. of variation was <18% (5 results) for each metal. M. P.

1B72. **Potentiometric and conductometric titration of mercury(II) salts and of mixtures of mercury(II) salts with cadmium, zinc or lead(II) salts with edta and dcta [1,2-diaminocyclohexane-*NNN′N′*-tetra-acetic acid] complexans.** Karlik, Milan; and Kout, Frantisek (Inst. Anal. Chem., Chem.-Tech. Hochsch., Prague, Czechoslovakia). *Sb. Vys. Sk. Chem.-Technol. Praze, Anal. Chem.*, 1977, **H11**, 65–81 (in German).—Potentiometric titrations were carried out with use of an amalgamated gold electrode and conductometric titrations with electrodes coated with platinum black. In the potentiometric methods, the best titrant was Na_4edta. With soln. >mM in Hg^{2+}, addition of the titrant caused pptn. of the Hg_2 - edta complex, and the potential change at the completion of this reaction was suitable for the determination of Hg^{2+}. Addition of further edta caused this ppt. to dissolve, with formation of the Hg - edta complex; a second potential change at the end of this reaction was suitable for the determination of down to 50μM-Hg^{2+}. Mixtures of Hg^{2+} with Cd^{2+} or Zn^{2+} showed two breaks, the first corresponding to the ppt. formation and the second to the sum of half the Hg^{2+} and the added cation. Titrations of mixtures of Hg^{2+} and Pb^{2+} were not successful. In the conductometric titrations, discontinuities in the curves corresponding to the two stages of titration were obtained. Similar determinations could be carried out of Hg^{2+} alone or in mixtures with Cd^{2+} or Zn^{2+}, when the best titrant was Na_2edta. G. R.

1B73. **Extraction of mercury by dithizone and stability of the extracted complex.** Litman, Robert; Williams, Evan T.; and Finston, H. L. (Dept. Chem., Univ., Lowell, Mass. 01854, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 983–987.—The extraction of Hg by a dithizone soln. in CCl_4, from aq. media containing a variety of solutes, was studied by tracer methods based on use of $^{203}Hg(NO_3)_2$ and by visible-region spectrophotometry. Results showed that extraction was incomplete at trace levels (0·1 μg ml^{-1}) of Hg, the complex was generally unstable, and extraction was hindered by the presence of some anions (*e.g.*, Br^- or I^-) at concn. normally encountered in environmental samples. R. W.

Group III

B, Al, Ga, In, Tl, Sc, Y, lanthanoids, actinoids

1B74. **Amperometric titration of boric acid with use of rotating tantalum and graphite electrodes.** Akhmedov, G.; and Zhdanov, A. K. *Uch. Zap. Dushanb. Gos. Pedagog. Inst.*, 1976, **99**, 118–121 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (17), Abstr. No. 17G138.

1B75. **Determination of boron and lanthanum in lanthanum boride.** Kobayashi, Michiko; Ichinose, Akio; and Einaga, Hisahiko. *Yogyo Kyokai Shi*, 1976, **84** (10), 513–520 (in Japanese).—The sample was dissolved in HCl, plus small amounts of HNO_3 to convert B into H_3BO_3, and an aliquot of the soln. was passed down a cation-exchange column to remove La. The H_3BO_3 was determined in the percolate by titrating with NaOH soln. in the presence of mannitol, with use of a pH meter to determine the end-point, and La was determined in another aliquot of the sample soln. by titration with edta soln. (xylenol orange as indicator). Excess of mineral acid present in the sample soln. was removed before the titration. CHEM. ABSTR.

1B76. **Long-chain ammonium salts as reagents in extraction - spectrophotometric determination of aluminium as a complex with catechol violet.** Tananaiko, M. M.; and Vdovenko, O. P. (T. G. Shevchenko Kiev State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1121–1126 (in Russian).—The Al is extracted into butanol as the Al - catechol violet - hexadecylpyridinium (1:2:5) complex from aq. medium of pH 7·4 to 10 (containing 50-fold and 5-fold molar amounts of hexadecylpyridinium iodide and catechol violet, respectively, relative to Al). The complex exhibits max. absorption at 594 nm ($\epsilon = 65{,}000$), and Beer's law is obeyed for 0·03 to 0·6 μg ml^{-1} of Al. M. P.

1B77. **X-ray fluorescence analysis of the alumina - silica system.** Mizuno, Kohichi; and Shiio, Hajime (Munic. Ind. Res. Inst., Nagoya, Japan). *Bunseki Kiki*, 1977, **15** (4), 172–180 (in Japanese).—The 'mineral effect' produced by differences in the chemical composition and crystal structure of sample particles, and which causes scatter in the results of X-ray fluorescence analysis, can be avoided by preparing the sample from a gel and then heating it, rather than by simple grinding of the solid components and heating, or by grinding the solid to give particles of diam. <1 to 2 μm. Thus, Al_2O_3 - SiO_2 (1:2) was prepared by mixing gels obtained by making $AlCl_3$ soln. alkaline (aq. NH_3) and making sodium silicate soln. acid (HCl), drying the mixture at 105° for 24 h, then heating it at 600° to 1700°. The X-ray fluorescence intensity for such samples remained constant; samples prepared by grinding the solid components showed fluctuations in fluorescence intensity with heating over this range. However, samples obtained by grinding the solids to a diam. of <1 to 2 μm showed no scatter in intensity values. CHEM. ABSTR.

1B78. **Determination of boron in aluminium and its alloys by flame emission spectroscopy.** Patricot, M. (Centre Rech. Aluminium Pechiney, Voreppe, France). *Analusis*, 1977, **5** (5), 236–241 (in French).—The sample (1 g) is dissolved in 20 ml of 6M-HCl (plus HNO_3 for certain alloys) and, after dilution, the soln. is extracted with $CHCl_3$ - 2-ethylhexane-1,3-diol (**I**) (9:1). In the extract, B, as its complex with **I**, is determined with use of a H - N_2O flame and measurement at 518 nm. The method is applicable in the range 0·001% to 5% of B in the original sample; for 0·006% and 1% of B the relative error was ±12% and 1%, respectively. B. D. H.

1B79. **Precipitation in homogeneous solution. III. Precipitation of the 8-hydroxyquinoline complex of gallium in basic medium.** Espinosa Boissier, A.; and Utrilla Sanchez, M. P. (Dept. Quim. Anal., Fac. Cienc. Quim., Univ. Complutense, Madrid, Spain).

Quim. Anal., 1977, **31** (1), 37–41 (in Spanish).—The Ga-containing soln. is treated with 60 ml of acetone, 30 ml of H_2O, sufficient 8-hydroxyquinoline soln. in acetone (1 g in 9 ml) to precipitate the Ga and ammoniacal buffer soln. of pH 9·8; the mixture is heated at 80° to volatilise the acetone and precipitate the Ga complex. The ppt. is collected, washed with H_2O containing a few drops of the buffer soln., dried for 3 h and weighed. The residual soln. is treated with 1% 8-hydroxyquinoline soln. in $CHCl_3$, and the absorbance of the separated $CHCl_3$ layer is measured at 392 nm to check that pptn. of Ga is complete. A thermogravimetric study of the ppt. is reported. L. A. O'N.

1B80. **Highly sensitive spectrophotometric determination of gallium with Pontachrome azure blue B [C. I. Mordant Blue 1] and hexadecyltrimethylammonium chloride.** Uesugi, K.; and Shigematsu, T. (Lab. Chem., Inst. Technol., Himeji, Japan). *Talanta*, 1977, **24** (6), 391–393.—Gallium forms a 1 : 4 complex with Pontachrome azure blue B (**I**) in the presence of hexadecyltrimethylammonium chloride (**II**); $\epsilon = 1{\cdot}39 \times 10^5$ at 680 nm and the spectrophotometric sensitivity is 0·49 ng cm^{-2} of Ga. *Method*—To a soln. containing 2 to 15 μg of Ga is added 3mM-**II** (1 ml), 0·1% aq. **I** (1 ml), 0·3M-HNO_3 - Na acetate buffer of pH 6·5 (5 ml) and H_2O to 20 ml. The mixture is heated to 60° to 70° for 10 min, cooled and diluted to 25 ml, and the absorbance is measured at 680 nm. There is interference from Cu^{II} and Fe^{III}, but Al, Be, Sc, Th and U^{VI} can be masked with F^- or PO_4^{3-}. T. J. S.

1B81. **Co-extraction in the chlorogallate anion - basic dye - organic solvent system.** Tarayan, V. M.; and Pogosyan, A. N. (State Univ., Erevan, Arm. SSR). *Arm. Khim. Zh.*, 1976, **29** (10), 855–860 (in Russian).—The extraction - spectrophotometric determination of micro amounts of Ga associated with basic dyes has been studied. An aq. soln. of $GaCl_3$ containing H_2SO_4 and NaCl or LiCl, was mixed with an aq. soln. of methyl green, and the ion-associate was extracted with an equal vol. of organic solvent. The absorbance of the organic phase was measured at 630 to 635 nm. The effects of H_2SO_4 concn. (7 and 5·5M and pH 1), Cl^- concn. (0·9 to 10M), dye concn. (up to 0·5mM), and solvent (benzene or benzene - dichloroethane or ethyl acetate mixtures) on the absorbance are illustrated. Conditions leading to the co-extraction of the dye are indicated. A. S. W.

1B82. **Reduction of a molybdotungstogallate complex [and spectrophotometric determination of gallium].** Tsyganok, L. P.; and Sukhova, N. S. (Dnepropetrovsk State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1103–1107 (in Russian).—A Ga - Mo^{VI} - W^{VI} (1 : 6 : 6) heteropoly-complex is formed at pH 4 to 5 (in the presence of ≃80-fold molar amounts of Mo and W relative to Ga) when the reaction mixture is heated at 100° for 15 min. The complex is reduced by Na_2SO_3 (by heating the mixture, containing ≃10% of Na_2SO_3, at 100° for 3 min) to the blue complex species $GaMo^{V}Mo^{VI}_5W^{VI}_6O_{40}^{6-}$, which exhibits max. absorption at 582 nm. Beer's law is obeyed for 0·12 to 16mM-Ga. Molybdotungstate and Al do not interfere, but tartaric, citric and oxalic acids, AsO_4^{3-}, PO_4^{3-} and SiO_3^{2-} do. The coeff. of variation was <5·5% (5 results) when determining 0·35 to 1·75 mg ml^{-1} of Ga in prepared soln. containing 7-fold amounts of Al. M. P.

1B83. **Complexometric determination of gallium and gadolinium in gadolinum gallium garnet crystals.** Novak, Josef (Phys. Inst., Acad. Sci., Prague, Czechoslovakia). *Fresenius' Z. Anal. Chem.*, 1977, **285** (2), 126 (in German).—The total Ga plus Gd content is determined by adding excess of edta and back-titrating with $ZnSO_4$ soln. at pH 6·5 to 8·5 with Eriochrome black T (C.I. Mordant Black 11) as indicator. The soln. is adjusted to pH 4 with acetic acid, NaF is added, and the soln. is boiled to decompose the Gd complex. Sodium acetate (to give pH 5 to 6) and xylenol orange are added and the liberated edta is titrated with $ZnSO_4$ soln. For prepared soln. the standard deviations were 0·18 and 0·12%, respectively, for determinations of Ga and of Gd. D. B. A.

1B84. **Fluorimetric determination of indium with 8-mercaptoquinoline.** Watanabe, Kunihiro; Fujiwara, Akio; and Kawagaki, Kyozo (Fac. Sci. and Technol., Sci. Univ. Tokyo, Noda-shi, Chiba, Japan). *Bull. Chem. Soc. Jpn.*, 1977, **50** (6), 1460–1463.—Acetic acid (10 ml) and 0·2% 8-mercaptoquinoline soln. (0·2 ml) are added to the test soln. (containing 0·5 to 25 μg of In), the pH is then adjusted to 2·5, the mixture is diluted to 50 ml, and the In complex is extracted into $CHCl_3$ or isobutyl methyl ketone (10 ml) for fluorimetry at 515 nm, with excitation at 365 nm. Of 29 cations studied, those of Ga, Zn and Cd caused positive errors, and those of Co, Cu, Fe, Ni, Pd, Hg, Bi, Sb, Au and Ag reduced the fluorescence. Interference by up to 1 mg of Fe, 100 μg of Hg or 1 mg of Ag can be masked by using ascorbic acid and KI plus thiourea. E. A.

1B85. **Biamperometric titration of indium and copper with magnesium - edta in non-aqueous medium.** Gevorgyan, A. M.; Talipov, Sh. T.; Molodozhen, E. V.; Kostylev, V. S.; and Khadeev, V. A. *Uzb. Khim. Zh.*, 1977, (2), 80–82 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (17), Abstr. No. 17G89.

1B86. **Extraction and spectrophotometric determination of thallium as the tetrabromothallate of pyronine B [6-diethylamino-3*H*-xanthen-3-ylidene-ammonium chloride].** Naruskevicius, L.; and Biryukova, Z. V. *Liet. TSR Aukst. Mokyklu Mokslo Darb., Chem. Chem. Technol.*, 1976, (18), 15–20 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G124.

1B87. **Luminescence determination of scandium with use of phenyl salicylate.** Bel'tyukova, S. V.; Drobyazko, V. N.; and Kononenko, L. I. (Inst. Gen. and Inorg. Chem., Acad. Sci. Ukr. SSR, Kiev). *Ukr. Khim. Zh. (Russ. Ed.)*, 1977, **43** (6), 641–644 (in Russian).—Phenyl salicylate forms with Sc a stable suspension of a poorly soluble ppt., which exhibits dark-blue luminescence in u.v. radiation. The sample (mixed rare-earth compounds) is dissolved in HCl, and the soln. is diluted with H_2O to give a concn. of 1 mg ml^{-1} of M_2O_3. A portion (0·5 ml) of this soln. is mixed with ethanolic 0·01M-phenyl salicylate (0·6 ml) and 1 ml of hexamine buffer soln. of pH 5·5, the mixture is diluted to 5 ml with H_2O, and the luminescence at 450 nm is measured after 30 min. The Sc content is determined by the method of additions. There is no interference from up to 100 μg of alkali or alkaline-earth metals, Be, Pt, Cd, Tl, Bi or Mn, or up to 30 μg of Zn or Ni. The luminescence is completely quenched by Al, V, W or Fe, and is halved by 30 μg of Ge, Sn, Pb, Nb,

Sb, Se or Cu. Of the rare-earth metals, only Eu and Er reduce the luminescence (by <20%), and errors caused by the presence of Y are <3%.
A. B.-S.

1B88. **Ion-exchange pre-concentration of elements of the cerium sub-group from 'pure' yttrium [for spectrographic determination].** Ryabukhin, V. A.; Gatinskaya, N. G.; and Ermakov, A. N. (V. I. Vernadskii Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (5), 909–913 (in Russian).—The sample (1 g) was evaporated with 20% HCl, and the residue was evaporated several times with H_2O to yield 5 ml of test soln. of pH 2 to 3. This soln. was applied to a column (44 cm × 5 cm) of KU-2 resin (pre-treated with 0·25M-citric acid soln. adjusted to pH 2·86 with aq. NH_3). The Y^{III} (≃95% of the amount present) was eluted with 0·25M-citric acid of pH 2·86 (1·9 l), and the La, Ce, Pr and Nd (with the remainder of the Y^{III}) were eluted with the same eluent (2·8 l). The latter eluate was passed through a column (13 cm × 0·9 cm) of KU-2 resin (H^+ form) and the Y^{III} and lanthanoids were eluted with 4M-HCl (50 ml). This eluate was evaporated to dryness, the residue was dissolved in H_2O (10 ml), the lanthanoids were pptd. as oxalates, and these were converted into oxides for determination by conventional spectrography. The method has been used in determining La (18 p.p.m.), Ce (8), Pr (≃10) and Nd (26); analysis took ≃14 h.
M. P.

1B89. **Complexometric determination of total rare-earth metals in mineral concentrates and products of their processing.** Afonina, T. A.; Kondrat'eva, T. M.; Merisov, Yu. I.; Norina, T. M.; and Petrova, E. I. (State Sci.-Res. and Design Inst. for Rare-Metal Ind., Moscow, USSR). *Zavod. Lab.*, 1977, **43** (6), 644–646 (in Russian).—Dissolve the sample (200 mg) by heating with 10 ml of HCl - HNO_3 (3 : 1), adding 1·5 ml of HF - H_3BO_3 (2 : 1) and 3 ml of conc. H_2SO_4 to complete the dissolution. Heat to fumes, and evaporate an aliquot containing ≃2 mg of rare-earth metals to a moist residue; add conc. H_2SO_4, and again heat to fumes. Add 2 ml of 50% $Al(NO_3)_3$ soln. to the dry residue, then add 8 to 10 drops of H_2O_2 soln. and 5 ml of HCl (1 : 1), and boil. Add ≃80 ml of H_2O, boil, and adjust the pH of the hot soln. to ≃2 by adding dil. NH_3; then add 20% hexamine soln. dropwise to give pH 4 to 4·5. Heat to coagulate the ppt., then add 2 ml of 0·2% polyacrylamide soln. dropwise, and heat to yield a flocculent ppt. Cool, dilute to 100 ml with H_2O, filter, and to 10 to 50 ml of filtrate add 5 ml of 25% sulphosalicylic acid, ≃10 mg of ascorbic acid, 4 to 6 drops of 0·1% 2,6-dinitrophenol soln., and adjust the pH to 4·5. Add acetate buffer soln. (pH 5·4), heat at ≃90° for ≃5 min, and dilute with ≃120 ml of H_2O. Add 3 to 5 drops of arsenazo III soln., and titrate the total rare-earth metals with 0·01M-edta to a violet end-point.
A. S. W.

1B90. **Determination of lanthanum in mixed rare-earth oxides by flame photometry.** Gim, Nae Soo; and Li, Byung Tae. *Punsok Hwahak*, 1976, **14** (3), 128–130 (in Korean).—To determine La in a typical sample (CeO_2 40 to 50%; La_2O_3 20 to 25%; Nd_2O_3 5 to 10%; and Pr_2O_7 2 to 3%), Ce (which interferes) was removed by pptn. as iodate, and the other lanthanoids were pptd. as bromates. The ppt. was ignited at 800°, and a soln. of the residue in HCl (1 : 1) was evaporated to dryness. Ethanol and H_2O were added, the pH of the resulting soln. was adjusted to 6, and the transmittance was measured at 563 nm by using an acetylene - air flame. The procedure was repeated with a standard La soln. The optimum range of the method was 2 to 20 mg ml^{-1} of La in 50% chloride soln. in ethanol; Nd did not interfere.
CHEM. ABSTR.

1B91. **Fumaric acid and the precipitation of cerium(III).** Maghzian, R. (Dept. Chem., Fac. Sci., Univ. Isfahan, Iran). *Radiochem. Radioanal. Lett.*, 1977, **30** (1), 53–59.—The ppt. obtained by adding Na fumarate to a Ce^{III} soln. at pH 4 to 5 is suitable for preparation of solid radioactive sources for counting, but pptn. is incomplete (owing to dissolution in excess of fumarate) and the H_2O content of the ppt. varies from nil to $6H_2O$ according to the temp. of drying. The Ce content should therefore be determined by, *e.g.*, titration with edta (xylenol orange as indicator).
J. W. P.

1B92. **Spectrophotometric determination of cerium with methyl green.** Jercan, Elena; Musat, Cristina; and Mainerici, Maria. *Rev. Chim.* (*Bucharest*), 1977, **28** (5), 470–471 (in Romanian).—Mix a soln. of $Ce(SO_4)_2$ in M-H_2SO_4 with 0·5 ml of 0·2% methyl green soln. in M-H_2SO_4, dilute to 10 ml with the acid, and measure the absorbance at 470 nm in a 1-cm cell against H_2O; obtain the Ce content from a calibration graph. Beer's law is obeyed for 1 to 11 μg ml^{-1} of Ce. To determine Ce in optical glass, dissolve the sample in H_2SO_4 - HF, and treat the soln. as described above. For small amounts of Ce, concentrate the soln. by evaporation or by passage through a cation-exchange resin (*e.g.*, Amberlite IR-120), followed by elution with M-H_2SO_4. There is no interference from other elements, except those that form a ppt. in H_2SO_4, but strong oxidising agents interfere. To determine Ce^{III}, oxidise the element to Ce^{IV}, remove unconsumed oxidant, and proceed as before.
C. D. K.

1B93. **Complex formation of neodymium, holmium and erbium with ethylenediaminetetra-acetic acid and catechol or pyrogallol.** Poluektov, N. S.; Tishchenko, M. A.; and Zheltvai, I. I. (Inst. Gen. and Inorg. Chem., Acad. Sci. Ukr. SSR, Odessa Lab.). *Ukr. Khim. Zh.* (*Russ. Ed.*), 1977, **43** (4), 407–411 (in Russian).—Mixed-ligand 1 : 1 : 1 complexes were obtained when 0·1M-$NdCl_3$, $HoCl_3$ or $ErCl_3$ (1 ml) and edta (1 ml) were mixed with 0·1M-catechol or pyrogallol (1·5 ml) together with H_2O to produce 9·7 ml, and the mixture was covered with a layer of xylene, then made alkaline by adding 0·3 ml of 0·1M-$NaBH_4$ (to prevent oxidation) in 2M-NaOH. The absorption spectra showed sharp peaks between 16,000 and 18,000 cm^{-1} with Nd, 21,000 and 23,000 (and 27,000 and 28,000 cm^{-1}) with Ho and 18,000 and 20,000 (and 25,000 and 27,000 cm^{-1}) with Er. The method of formation and the structure of the complexes are discussed.
G. S. S.

1B94. **Determination of individual rare-earth metals in samples of mixed salts.** Skorova, I. D.; Samsonova, N. I.; and Marushko, A. V. *Zavod. Lab.*, 1977, **43** (6), 684–685 (in Russian).—The X-ray spectrographic determination of individual rare-earth metals in complex mixtures with use of *L*-series lines is difficult; in the simple rapid method proposed, *K*-series lines are measured after preliminary separation of the sample into fractions containing only two or three metals. An instrument with a tungsten target is suitable. The method has been used for the determination of Eu, Sm and Gd (total rare-earth metals

20 to 30 g l^{-1}) in HNO_3 medium (300 to 400 g l^{-1} of acid) and for that of Nd (20 to 30 g l^{-1}) and Gd (0·05 to 0·3 g l^{-1}) in technical soln. A. S. W.

1B95. **Selective spectrophotometric determination of uranium.** Budesinsky, B. W. (Phelps Dodge Corp., Morenci, Ariz. 85540, U.S.A.). *Microchem. J.*, 1977, **22** (1), 50–54.—To mask interfering ions, a soln. (100 ml) containing U^{VI} (10 to 100 μg) is mixed with 1,2-diaminocyclohexane-*NNN'N'*-tetra-acetic acid (dcta) (420 mg per mmol of interfering ion) and adjusted to pH 6 to 7 with aq. NH_3. The soln. is extracted with dibenzoylmethane (2% in benzene), the organic phase is washed with 0·01M-Na_2dcta and extracted with 6M-HCl, and the acid extract is washed with benzene. The acid soln. is evaporated to dryness, the residue is dissolved in 0·1M-HCl, and the U^{VI} is determined with arsenazo III at 650 nm. The average deviation is $< \pm 0{\cdot}1$ μg. The effects of 30 interfering ions on the determination are listed. T. G. A.

1B96. **Spectrophotometric study of the reaction of uranium(VI) with bromopyrogallol red, and determination of uranium(VI).** Sucmanova-Vondrova, M.; Havel, J.; and Sommer, L. (Dept. Anal. Chem., J. E. Purkyne Univ., Brno, Czechoslovakia). *Collect. Czech. Chem. Commun.*, 1977, **42** (6), 1812–1829.—A study is presented of the mechanism of complex formation in the system bromopyrogallol red (**I**) - U^{VI} - aq. ethanol. In the presence of quaternary-base cations, ternary complexes with relatively high ε values are formed. Three procedures for determining U are described. (*i*)—To the weakly acid test soln. containing <1 mg of U^{VI} are added 5 ml each of a **I** soln. (prep. described), M-pyridine buffer of pH 6·05 and masking reagent [containing $Ca(NO_3)_2$, Na_2edta, Na 5-sulphosalicylate and K Na tartrate]; the mixture is diluted to 50 ml with H_2O, and the absorbance is measured at 630 to 680 nm. (*ii*)—To the weakly acid test soln. containing <375 μg of U^{VI} are added 5 ml each of ethanolic 0·5% **I**, M-pyridine buffer of pH 6 to 7 and aq. 2% poly(vinyl alcohol) and 10 ml of 25mM-butyltriphenylphosphonium bromide (**II**) in 25% ethanol. The soln. is then diluted to 25 ml with H_2O, and, after 10 min, the absorbance is measured at 650 nm. (*iii*)—As in method (*ii*), but with pyridine buffer soln. of pH 5·9 and ethanolic 60mM-hexadecylpyridinium bromide in place of **II**. Carbonate, HPO_4^{2-}, F^- and WO_4^{2-} interfere seriously. J. M. B.

1B97. **Determination of uranium in 'real' samples of ammonium diuranate.** Krtil, J.; Vachuska, J.; Spevackova, V.; Kuvik, V.; and Lukavsky, J. (Nucl. Res. Inst., Rez, Prague, Czechoslovakia). *Radiochem. Radioanal. Lett.*, 1977, **30** (1), 7–11.—It was found by t.g.a. that a suspension of ammonium diuranate in water containing ammonium salts and free NH_3 can be dried at 50° without decomposition. The dry product, after being ground, is dissolved in 3M-HNO_3, and U is reduced with $FeSO_4$ soln.; the soln. is then treated with 0·05M-$VOSO_4$, and the U^{IV} is titrated with 0·1N-$K_2Cr_2O_7$. J. W. P.

1B98. **Parts-per-million determinations of uranium and thorium in geological samples by X-ray spectrometry.** James, Gerard W. (Geol. Survey, Univ. Kansas, Lawrence, Kans. 66044, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 967–969.—Samples (10 g) were analysed directly as powders (<200 mesh) by measuring the *L*α radiation emitted by U and Th on excitation with X-rays from a molybdenum target. From 0 to 100 p.p.m. of U and Th could be determined rapidly and accurately, and, with a 5-min counting time, the detection limits were 1·2 and 1·5 p.p.m., respectively. By calibrating with standards prepared from USGS igneous rocks, Ottawa sand and carnotite ore, interference from other elements (*e.g.*, Rb and Sr) was negligible. For the determination of up to ≃50 p.p.m. of U in sandstone, the results obtained agreed, on average, to within 4 p.p.m. of those of a conventional fluorimetric method. R. W.

1B99. **Acetylacetone as chelating reagent, extracting solvent and electrolysis medium: polarographic determination of uranium(VI) and iron(III).** Fujinaga, Taitiro; and Lee, Heung Lark (Dept. Chem., Fac. Sci., Kyoto Univ., Japan). *Talanta*, 1977, **24** (6), 395–396.—Acetylacetone (**I**) was fractionally distilled over $NaHCO_3$ and P_2O_5 before use as a polarographic medium. The accessible potential ranges of **I** are from −0·16 to −2·26 V *vs.* a silver - 0·1M-$AgClO_4$ electrode for pure **I**, and from −0·35 to −2·20 V for **I** saturated with H_2O. In pure **I**, the Fe^{III} complex gives one wave ($E_{\frac{1}{2}}$ = −1·43 V) and the U^{VI} complex gives two waves ($E_{\frac{1}{2}}$ = −1·55 and −1·90 V). After extraction from aq. soln. at pH 6·8 to 7·0, both metal acetylacetonates are reduced more reversibly ($E_{\frac{1}{2}}$ for Fe^{III} = −1·29 V, $E_{\frac{1}{2}}$ for U^{VI} = −1·52 V). Calibration graphs are rectilinear for 10μM to mM concn. of metal ion in the extract. T. J. S.

1B100. **Perchloric acid dissolution of [irradiated] graphite and pyrolytic carbon [coatings on reactor-fuel particles].** Buzzelli, George; and Mosen, Arthur W. (General Atomic Co., San Diego, Calif. 92138, U.S.A.). *Talanta*, 1977, **24** (6), 383–385.—Safe procedures were developed for the dissolution of irradiated materials. The most vigorous procedure is for dissolution of graphite and pyrolytic carbon with $HClO_4$ - fuming HNO_3 in a Soxhlet extractor. In a second procedure, mg amounts of pulverised reactor-fuel particles are dissolved by heating with $HClO_4$ - HNO_3 on a sand bath. A third procedure consists of dissolution of more easily oxidised non-graphitic carbonaceous material in fuming HNO_3. The hazards of using $HClO_4$ in the presence of easily oxidisable materials are stressed. T. J. S.

1B101. **Solvent-extraction studies using tetracycline as complexing agent. VII. Separation of uranium from scandium and rare-earth metals with ethylenediaminetetra-acetic acid as masking agent.** Saiki, M.; and Lima, F. W. (Radiochem. Dept., Inst. Energ. At., Sao Paulo, Brazil). *Radiochem. Radioanal. Lett.*, 1977, **30** (1), 1–5.—Although U, Sc, La, Eu and Tm are all extracted from aq. soln. by 0·01M-tetracycline in benzyl alcohol, only U is extracted in the presence of edta at pH 2·9. (For Part VI see *Anal. Abstr.*, 1977, **33**, 5B85.) J. W. P.

1B102. **Plutonium determination by extraction spectrophotometry in spent fuel from a nuclear-power plant.** Sebesta, F.; Sedlacek, J.; and Podhajecky, P. (Katedra Jad. Chem., Vys. Uceni Tech., Prague, Czechoslovakia). *Jad. Energ.*, 1976, **22** (8), 301–303 (in Czech).—In the method described, Pu^{IV} is quantitatively and selectively separated by extraction into 0·5M-2-thenoyltrifluoroacetone in xylene from a medium M in HNO_3 and re-extraction into 10M-HNO_3 for spectrophotometry (with arsenazo III) at 665 nm. For an average value of 3·5 μg of Pu in 1 mg of uranium, the standard deviation of an individual determination was ±65 ng. The results agreed well with those calculated by two independent methods. INIS Atomindex

1B103. **Possible use of alpha-spectrometry for analysis of isotopic composition of plutonium in nuclear-fuel elements.** Rodionov, Yu. F.; Shatinskii, V. M.; and Zelenkov, A. G. (I. V. Kurchatov Inst. At. Energ., Moscow-Oblinsk, USSR). *Isotopenpraxis*, 1976, **12** (7–8), 271–276 (in Russian).—The α-particle spectrum from Pu separated chemically from nuclear-fuel elements is recorded in the energy range 4·5 to 5·7 MeV with a semiconductor spectrometer having a gold - silicon detector to obtain peaks for ^{238}Pu, ^{239}Pu plus ^{240}Pu, and ^{241}Pu plus ^{242}Pu. A magnetic spectrograph having a spatially sensitive semiconductor detector is then used in the range 5·14 to 5·18 MeV to determine the ratio of activities due to ^{239}Pu and ^{240}Pu, and the relative proportions of ^{238}Pu, ^{239}Pu, ^{240}Pu and ^{241}Pu are calculated (the contribution of ^{242}Pu is ignored). In an example, the contents and relative errors (%) for the isotopes cited are, respectively, 0·11, ±0·36; 84·0, ±2·7; 12·3, ±2·8; and 3·6, ±15. C. D. K.

1B104. **Spectrophotometric titration of thorium and zirconium with edta using some halogenated phenylazochromotropic acid dyes as indicators.** Khater, M. M.; Issa, Y. M.; and Badawy, S. S. (Chem. Dept., Fac. Sci., Cairo Univ., Giza, Egypt). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 45.—The 3-(3-chlorophenylazo)-derivative of chromotropic acid (used at pH 4 and with measurement at 585 nm) gave best results for Zr (36 to 103 μg), and the 3-(2,4-dichlorophenylazo)-derivative (at pH 3·5 and 595 nm) the best results for Th (46 to 160 μg). Successive amounts of mM-edta, 2 ml of mM-indicator and 5 ml of hexamine buffer soln. of appropriate pH in 10 ml of H_2O are used, and the absorbance is measured immediately. The 3-(4-chloro-, 4-bromo- and 4-iodo-phenylazo)-derivatives can also be used as indicators. H. L. L.

1B105. **Determination of plutonium/uranium ratios by controlled-potential coulometry at a solid electrode.** Phillips, G.; and Crossley, D. (Chem. Div., AERE, Harwell, Oxon., England). *U.K. At. Energy Auth. Rep.*, AERE-R 8794, 1977. Pp. 23.—In the study described, U and Pu, in M-HCl or M-H_2SO_4, were oxidised and reduced in a cell fitted with a gold or platinum electrode. The U and Pu, respectively, were first reduced to U^{4+} and Pu^{3+} and then, after the elimination of adsorbed H, were oxidised to UO_2^{2+} and Pu^{4+}, the latter ensuring the rapid oxidation of the U. The Pu was then reduced and re-oxidised. From the values of the time - current integrals the contents of U and Pu were calculated, the results with M-H_2SO_4 having better precision than those with M-HCl. Details of the electrolysis cell and method of working are given. G. J. H.

1B106. **Separation of americium and iron on cationic resin in diethylenetriaminepenta-acetic acid medium.** Bergey, C.; and Ravenel, J. (C.E.A., Centre d'Etudes Bruyeres-le-Chatel, Montrouge, France). *Analusis*, 1977, **5** (5), 215–219 (in French).—The separation of Am from Fe on a column of AG 50W-X8 resin (H^+ form; 100 to 200 mesh) was examined and distribution coeff. were determined as a function of pH and concn. of diethylenetriaminepenta-acetic acid (DTPA). Americium was preferentially adsorbed, at pH 1·3, when the ratio of concn. of DTPA to Fe was >2, and it could then be eluted (up to 99% recovery) with 6M-HNO_3, after washing the column with 10mM-DTPA (ammonium salt). The effects of other ions are discussed. B. D. H.

Group IV

C, Si, Ge, Sn, Pb,
Ti, Zr, Hf

1B107. **Application of NAS 11–18 electrode to argentimetric micro-titration of cyanide.** Akimoto, Naoshigo; Ozaki, Motoaki; and Hozumi, Keiichiro (Fac. Pharm. Sci., Univ., Sakyo-ku, Kyoto, Japan). *Bunseki Kagaku*, 1977, **26** (6), 417–419 (in Japanese).—The Na^+-selective glass electrode (Corning) has been used as an end-point detector in the automatic titration of CN^- with Ag^+. The pH of an aliquot of 5mM-KCN, 0·01M in KOH, was adjusted to 10·5 with K_2CO_3 - $KHCO_3$ buffer and the soln. was made up to 50 ml. The soln. was titrated with 5mM-$AgNO_3$ with use of a double-junction s.c.e. and the NAS 11–18 Na^+-selective glass electrode as indicator electrode. The first potential jump, corresponding to the formation of $Ag(CN)_2^-$, was used in the determination. Titration should be carried out within 10 min to avoid decomposition of the CN^- soln.

1B108. **Analysis of carbon powder by emission spectroscopy in an arc stabilised by a magnetic field.** Gorbunova, L. B.; Avdeenko, M. A.; Kuteinikov, A. F.; and Volod'kina, T. V. *Zavod. Lab.*, 1977, **43** (6), 686–687 (in Russian).—Use of a discharge stabilised by a non-uniform magnetic field (≃14,000 A m^{-1}) produces enhancement of the intensity of the lines for Fe, Al, Mg and Si at 248·3, 308·2, 279·5 and 251·6 nm, respectively, and permits determinations to be made without use of NaCl as carrier. The time required for a determination is thus considerably reduced, and errors arising from contamination during sample preparation are minimised. A. S. W.

1B109. **Spectral analysis of semiconductor silicon for impurities of hard-to-excite elements in a hollow-cathode discharge.** Rudnevskii, N. K.; Pichugin, N. G.; and Maksimov, D. E. *Zh. Prikl. Spektrosk.*, 1976, **25** (5), 921–923 (in Russian).—Bromine and Cl were determined in the sample by emission spectrography with use of a 0·6-A hollow molybdenum cathode He discharge at 481·7 and 476·6 nm, respectively, and an exposure time of 180 s. The sensitivity of the method was 3 p.p.m., and the mean coeff. of variation was 10%, for both elements, when purified He and a combined pulse - stationary current-generation mode were used.

CHEM. ABSTR.

1B110. **Quantitative Auger analysis of ion-implanted boron and arsenic in polycrystalline silicon.** Fujiwara, K.; Ohtani, M.; Kanayama, K.; and Ogata, H. (Central Res. Lab., Mitsubishi Electric Corp., Amagasaki, Hyogo, Japan). *Surf. Sci.*, 1976, **61** (2), 435–442.—Impurities of B and As implanted in the Si film (≃1·3 μm thick) at energies of up to 350 keV (with a total dose of up to 10^{16} atoms cm^{-2}) were determined semi-empirically. In-depth concn. profiles were obtained by sputter etching. The rectilinear relationship established between concn. and Auger signal intensity, normalised to the Si *LVV* transition of 92 eV, corresponded to the B and As concn. within 10% and 20%, respectively.

M. A. P.

1B111. **Quantitative secondary-ion mass-spectrometric analysis of oxygen isotopes and other light elements in silicon oxide films.** Croset, M.; and Dieumegard, D. (Lab. Central Rech. Thomson, C.S.F., Orsay, France). *Corros. Sci.*, 1976, **16** (10), 703–715.—Calibration graphs were obtained for ^{11}B,

^{16}O and ^{19}F by comparison of the results obtained for standard samples, prepared either by anodic oxidation or by ion implantation, with those of nuclear micro-analysis; charging effects were avoided by use of an aluminium grid on the surface of the sample. The sensitivity of the method was $\simeq 1\%$ isotopic concn. for ^{16}O, 10^{18} atoms cm^{-3} for ^{19}F, and 10^{16} atoms cm^{-3} for ^{11}B. The method was used to study the incorporation and movement of B, O and F in an amorphous thin film of silica during its anodic growth, and the results are discussed in terms of the mechanisms of anodic oxidation of silicon.

CHEM. ABSTR.

1B112. **Group radio-chromatographic separation of trace impurities in neutron-activation analysis of special-purity silica.** Ivanova, M. M.; Ogloblina, I. P.; Genel', S. A.; Mitina, V. V.; Kalinin, A. I.; and Lambrev, V. G. *Zh. Anal. Khim.*, 1977, **32** (6), 1066–1070 (in Russian).—To separate μg amounts of the impurities into groups suitable for γ-ray spectrometry, the test soln., made 10mM in HF, was applied to a column of KU-2X8 cation-exchange resin (H^+ form); Sb, Mo, As and W were eluted with 10mM-HF (fraction *A*); Fe, Sc and Cr with 0·5M-HF; Ga, In, Na, K and Cs with 5M-HF; and Ni, Co, Cu, Mn, La, Zn, Ag and Cd with 4M-HCl (fraction *B*); Au was retained on the column. Fraction *A* was evaporated to dryness, and a soln. of the residue in 8M-HCl - M-HF (1 : 1) was applied to a column of AV-17X8 anion-exchange resin (Cl^- form); As and W were eluted with 8M-HCl - M-HF (1 : 1), and Mo and Sb with 0·3M-HCl - M-HF (1 : 1) and then M-HCl. Fraction *B* was made 8M in HCl and applied to another column of AV-17X8 resin; Ni, Mn, La and Ag were eluted with 8M-HCl; Co and Cu with 3M-HCl; Zn with 20mM-HCl; and Cd with M-NH_3. The method was applied to silica containing 0·01 p.p.b. to 1 p.p.m. of the named elements; samples were irradiated in a flux of $\simeq 1{\cdot}2 \times 10^{13}$ n cm^{-2} s^{-1} for 70 h and subsequently decomposed by heating with HF - 6M-$HClO_4$ (1 : 1). M. P.

1B113. **Separation of matrix from impurities by heterogeneous fluorination [in spectrographic analysis of quartz].** Karyakin, A. V.; Semenova, L. F.; and Ozhegov, P. I. (V. I. Vernadskii Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (5), 926–932 (in Russian).—Impurities of Al, Mg, Mn, Fe, Cu, Ni, Cr, Ca, Zn and Ti (0·1 to 1 p.p.m.) were determined in synthetic quartz after separation of the matrix as SiF_4. A cavity in a graphite electrode was partly packed with NH_4F - $NaHSO_4$ (2 : 1) (400 mg) as fluorinating agent, the mixture was covered with a porous graphite membrane (1 mm thick) and the sample (100 mg) was placed on the membrane. The electrode was then electrically heated at 250° for 20 min, and the residue on the membrane was excited in a 10-A d.c. arc; a graphite counter-electrode having a cavity packed with graphite containing 5% of NaCl as radiation buffer and 1% of Co as internal standard was used. The lower limits of determination were 0·1 p.p.m. for Fe, Mn, Ni, Cr and Zn and 0·05 p.p.m. for Cu, Mg, Al, Ca and Ti. The coeff. of variation ranged from 20 to 25% (6 results) for Mn (0·15 p.p.m.), Cu (0·8), Cr (0·2) and Ni (0·1), and the results agreed with those obtained by a.a.s. and n.a.a. M. P.

1B114. **Chemical purification of silicon. IV. Further purification of trichlorosilane and improvement of analytical procedures for boron and phosphorus in trichlorosilane and high-purity silicon.** Lu, Cheng-Li; Liu, Kwei-Ning; and Chang, Chau-Ting (Dept. Chem., Natl. Tsing Hua Univ., Hsinchu, Taiwan). *Proc. Natl. Sci. Counc., Part 1* (*Taiwan*), 1976, **9**, 313–319.—Impurities of B and P were removed from trichlorosilane by adduct formation with Lewis bases and acids in a double-distillation apparatus; CuCl was used as scavenger for removal of P, and succinonitrile, 1,4-dinitrobenzene, chloro-(triphenyl)methane, and benzaldehyde were suitable Lewis bases for removal of B. To determine B and P in trichlorosilane, the absorbances of the B - curcumin complex in ethanol and of molybdovanadophosphoric acid in amyl alcohol were measured at 540 and 305 nm, respectively, after several pretreatment steps. To determine P in high-purity silicon by n.a.a., a 500-mg sample was irradiated in a flux of $\simeq 2 \times 10^{12}$ n cm^{-2} s^{-1} for 30 h and, after a 20-h decay period, was dissolved in HNO_3 - HF (with a phosphate carrier). The soln. was treated with $HClO_4$, HBr, H_2O and $(NH_4)_2MoO_4$ and was extracted with isobutyl alcohol, and the alcoholic phase was washed with H_2O and evaporated to dryness with dil. HNO_3; the residue was used for measurement of the ^{32}P β-ray activity. The detection limit was 0·04 p.p.m.; Cl and S interfered.

CHEM. ABSTR.

1B115. **Use of circulation of carrier gas in gas-chromatographic analysis of tetrachlorosilane.** Zueva, M. V.; Saglanskii, Yu. M.; and Radchenko, K. A. (Inst. Chem., Acad. Sci. USSR, Gor'kii). *Zh. Anal. Khim.*, 1977, **32** (5), 971–973 (in Russian).—The g.c. assembly used, in which carrier gas is circulated in a closed system, is described; $\simeq 5$ l of carrier gas is needed for an analysis. Impurities of CCl_4, $CHCl_3$, CH_2Cl_2 and chloro-, 1,1-dichloro- and 1,2-dichloro-ethane were determined on a column (2 m) packed with Polysorb 1 and operated at 130°, with propene as carrier gas (velocity 5 cm s^{-1}) and thermal-conductivity detection. The h.e.t.p. for 1,2-dichloroethane was 1·7 cm (compared with 2·1 cm when using He as carrier gas), and the Porter sensitivities (*cf. Anal. Chem.*, 1956, **28**, 290) were better for CCl_4, $CHCl_3$ and the dichloroethanes when using propene. M. P.

1B116. **Gas-chromatographic analysis of [technical-grade] silicon tetrachloride [tetrachlorosilane] for organic impurities.** Zueva, M. V.; Feshchenko, I. A.; and Nikolaeva, L. G. (Sci.-Res. Inst. Chem., Gor'kii State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1249–1251 (in Russian).—From 5 to 900 p.p.m. of CH_2Cl_2, $CHCl_3$, 1,1-dichloroethane (**I**) and 1,2-dichloroethane (**II**) are determined by g.l.c. of the sample (up to 2 mg) at 54° on a column (0·6 m × 4 mm) of 20% of diphenylamine on Spherochrome, with flame ionisation detection. The $SiCl_4$ reacts with diphenylamine to form a non-volatile compound that does not interfere in the separation. Limits of determination are 8, 7, 6·5 and 5 p.p.m. for CH_2Cl_2, $CHCl_3$, **I** and **II**, respectively; coeff. of variation were $<6\%$ (50 results). M. P.

1B117. **Rapid method for semi-micro analysis of silicate rocks and minerals by complexometry.** Guletskaya, E. S.; Zorkina, L. S.; and Gusev, G. M. *Tr. Inst. Geol. Geofiz. Akad. Nauk SSSR, Sib. Otd.*, 1977, (315), 98–105 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G177.

1B118. **Determination of hydrogen in silicates using the ion-beam spectrochemical analyser: application to hydrolytic weakening.** Tsong, Ignatius S. T.; McLaren, Alex C.; and Hobbs, Bruce E. (Dept.

Phys., Monash Univ., Clayton, Vic. 3168, Australia). *Am. Mineral.*, 1976, **61**, 921–926.—An intense, stable and near-monoenergetic beam of Ar ions, produced by a duoplasmatron source, bombards an area (2 mm diam.) of the sample target. Emitted radiation, after passing through two interference filters to isolate the 656·3-nm H line, is detected by a photomultiplier. As successive layers are sputtered, the concn. profile for H can be determined. A rectilinear calibration graph of intensity against the product of rate of wt. change and percentage of H has been constructed for a series of standards. From this, the H contents (0·03 to 0·23%) of a number of synthetic quartz and silicate samples have been determined (after heating at 130° to remove surface-adsorbed water); the values obtained are much higher than those determined by i.r. spectroscopy. M. A. P.

1B119. **Determination of fluorine in silicates by use of an ion-selective electrode following fusion with lithium metaborate.** Bodkin, J. B. (Miner. Constitution Lab., Pennsylvania State Univ., University Park, Pa. 16802, U.S.A.). *Analyst* (*London*), 1977, **102** (1215), 409–413.—Fuse 200 mg of sample (200 mesh) with 800 mg of anhyd. $LiBO_2$ in a graphite crucible at 1050° for 10 min, pour the melt into 80 ml of 4% HNO_3, and when dissolution is complete dilute to 100 ml. To 40 ml of this soln. add 10 ml of H_2O and 50 ml of a complexing buffer soln. (containing 9·1 g l^{-1} of 1,2-diaminocyclohexane-*NNN'N'*-tetra-acetic acid, 150 g l^{-1} of Na citrate dihydrate, and 30 g l^{-1} of NaCl; adjusted to pH 6·85) and determine F^- by means of an Orion 94–09 F^--selective electrode, with use of a standard-addition technique. It is important that an electric furnace be used for the fusion, as use of a gas burner leads to low results. Good agreement with accepted values was obtained for several standard rocks. R. W.

1B120. **Spectrophotometric determination of germanium by using antipyrine dyes.** Zhivopistsev, V. P.; and Cherepanova, T. B. (A. M. Gor'kii Perm State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 977–980 (in Russian).—The species $H[Ge(MO_3O_{10})_4]^{3-}$ (**I**) forms a 1:3 complex with antipyrinylbis-(4-dimethylaminophenyl)methanol (**II**). **I** is initially formed in 0·1M-HCl medium containing ≃0·3% of ammonium molybdate, and the **I** - **II** complex is formed in a medium 0·8M in HCl and containing an ≃7-fold molar excess of **II** (relative to Ge) and 0·02% of gelatin (as protective colloid). The **I** - **II** complex exhibits max. absorption at 610 nm (ϵ = 43,000), and Beer's law is obeyed for 0·1 to 1 μg ml^{-1} of Ge. There is no interference from the amounts (relative to Ge) shown in parentheses of the following species—Ba, Ca or Mg (5000), Co^{II} or Cs (500), Cu^{II}, Tl^{I} or Sr (200), Y, Ga, Cd, Se^{IV}, I^-, SCN^- or Br^- (100), Al, Be, Mn^{II} or Bi (50), La or In (30), Li, Sc, Ni or Sb^{V} (10), Cr^{III} or Fe^{III} (5), or NO_3^- (4). M. P.

1B121. **Flotation - spectrophotometric determination of germanium in industrial materials.** Flyantikova, G. V.; Korolenko, L. I.; Vinarova, L. I.; and Chekirda, T. N. (Inst. Gen. and Inorg. Chem., Acad. Sci. Ukr. SSR, Odessa). *Zh. Anal. Khim.*, 1977, **32** (5), 1028–1030 (in Russian).—Such a method (*cf. Anal. Abstr.*, 1976, **30**, 6B122) has been used for determining 0·5 to 350 p.p.m. of Ge in ferrophosphorus, metallurgical slags, phosphorites and products of their processing, zinc concentrates and boiler sediments; procedures for decomposing and dissolving the samples are described. Materials containing P are initially treated with hot H_2SO_4 - H_2O_2 soln. or HNO_3 - H_2O_2 soln. to remove Cl^- and sulphides; zinc concentrates are initially treated with aq. NH_3 - H_2O_2 soln., and slags and sediments are initially roasted with $Mg(NO_3)_2$ at 600°. The residues are dissolved in HNO_3 - HF - H_3PO_4 (1:1:1) in each instance. If the sample contains Mn, the Mn^{VII} formed is reduced to Mn^{II} with ascorbic acid. M. P.

1B122. **Determination of germanium in oxides, ores and alloys by chlorination and gas chromatography.** Iatridis, Basil; and Parissakis, George (Inorg. and Anal. Chem. Dept., Natl. Tech. Univ., Athens, Greece). *Anal. Chem.*, 1977, **49** (7), 909–911.—The sample (7·5 to 20 mg of powdered germania or germanite or small pieces of a Ge - Au alloy) and CCl_4 were heated in a sealed borosilicate-glass tube at 575° for 15 to 30 min. The tube was then broken, and the volatile compounds were transferred in a stream of N to a glass g.l.c. column (183 cm × 4 mm) packed with 20% of DC-550 on Celite 545 and operated at 80°, with thermal-conductivity detection. The peak for $GeCl_4$ was well separated from those of Cl_2, CO_2, CS_2, $COCl_2$ and CCl_4. Calibration was effected by injecting known amounts of $GeCl_4$ into the column and measuring the areas of the resulting peaks. The method could be applied to samples containing 1 to 99% of Ge, and the detection limit was 10 μg of Ge. W. C. D.

1B123. **Separation of tin [by extraction] with bis-(2-ethylhexyl) phosphate.** Tarasova, V. A.; Levin, I. S.; Bykhovskaya, I. A.; Rodina, T. F.; and Tresnitskaya, R. D. (Inst. Phys.-Chem. Principles Mineral Raw Mater. Process., Acad. Sci. USSR, Siberian Branch, Novosibirsk). *Zh. Anal. Khim.*, 1977, **32** (5), 981–984 (in Russian).—The test soln. (containing 1 to 100 μg of Sn^{IV} and mg amounts of such accompanying elements as Cu, Co, Ni, Mn^{II}, Zn, Pb, Cd, Fe^{III}, Bi, Sb^{III}, In, Ga, Tl, As^{III}, Cr^{III}, Ti and Zr) is made 3M in HBr, and mercaptoacetic acid is added to reduce Fe^{III} to Fe^{II} and Tl^{III} to Tl^{I}. The Sn^{IV} is then selectively extracted with 0·5M-bis-(2-ethylhexyl) phosphate in heptane, the aq. phase is discarded, and the extract is washed twice with 3M-HBr and twice with H_2O. The extract (1 vol.) is mixed with tributyl phosphate (2 vol.), and Sn is extracted into 4M-H_2SO_4 to give a soln. suitable for the spectrophotometric determination of Sn with catechol violet or 4-nitrophenylfluorone. The method has been used for determining 0·5 p.p.m. to 1·5% of Sn in indium. M. P.

1B124. **Potentiometric determination of oxygen in tin using a solid electrolyte.** Kitaev, A. D.; Kunin, L. L.; and Ozhegov, P. I. (V. I. Vernadskii Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (5), 995–998 (in Russian).—The method involves measuring the e.m.f. of a cell formed by the fused sample, a solid electrolyte (85% of ZrO_2 plus 15% of CaO) and nickel containing O. The sample is first mechanically cleaned, etched with hot conc. HCl for 10 min and washed with CCl_4, and the e.m.f. is measured at 900° and at a pressure of 0·133 mPa. The dependence of e.m.f. on O concn. can be used for determining 4 to 38 p.p.m. of O dissolved in tin. The coeff. of variation is <4% (3 to 8 results). M. P.

1B125. **Oxide films on sputtered tin - nickel alloy exposed to pure oxygen and air.** Sharma, S. P.; and Thomas, J. H., III (Bell Telephone Laboratories Inc., Columbus, Ohio 43213, U.S.A.). *Anal. Chem.*,

1977, **49** (7), 987–990.—Films formed on electroplated Sn - Ni (13 : 7), as an $\simeq 2{\cdot}5$-μm layer (sputter-cleaned in pure Ar) on quartz, on exposure to O and to air (at pressures from 10^{-4} to 760 Torr) were studied by electron spectroscopy; mass-change measurements were made by use of a plated quartz-crystal oscillator. Oxides of Ni and of Sn, as well as anhyd. $NiSnO_3$, were thereby detected in such films. R. W.

1B126. **Spectroscopic isotopic analysis of lead.** Shipilov, L. D. *Tr. Inst. Geol. Goefiz. Akad. Nauk SSR, Sib. Otd.*, 1977, (315), 53–57 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (17), Abstr. No. 17G157.

1B127. **Dalzin [1,6-diallyl-2,5-dithiobiurea] as an analytical reagent for estimation of trace quantities of bismuth in lead and nickel.** Navaratna, M. R.; and Iyer, C. S. P. (Anal. Chem. Div., Bhabha Atom. Res. Centre, Bombay, India). *Talanta*, 1977, **24** (6), 396–398.—Lead (0·5 to 2 g containing <50 μg of Bi) is dissolved in 10 to 20 ml of HNO_3 (1 : 1) and heated to fuming with $HClO_4$ (2 to 3 ml), and the residue is dissolved in H_2O; 0·02M-dalzin in acetone (1 ml) is added, and the soln. is diluted to 50 ml, adjusted to pH 2·5 with dil. aq. NH_3 and then extracted with $CHCl_3$ (10 ml). Nickel (0·3 to 1 g containing <50 μg of Bi) is decomposed with HCl and $HClO_4$. The residue is dissolved in H_2O and treated with a 10-fold excess of 50% Na citrate soln.; 0·02M-dalzin in acetone (1 ml) is added and the soln. is diluted to 50 ml. After adjusting the pH to 6·5 with dil. aq. NH_3, the soln. is extracted with $CHCl_3$ (10 ml). The absorbances of the $CHCl_3$ extracts are measured at 350 nm. The Sandell sensitivity is 0·04 μg cm^{-2}; Pd and Cu interfere, but 100 p.p.m. of Co is tolerated. T. J. S.

1B128. **Spectrophotometric determination of titanium(IV) with 3-hydroxy-2-methyl-1-phenyl-4-pyridone.** Vojkovic, V.; Tamhina, B.; and Herak, M. J. (Lab. Anal. Chem., Fac. Sci., Univ., Zagreb, Yugoslavia). *Fresenius' Z. Anal. Chem.*, 1977, **285** (3–4), 266–267.—Extract, for 1 to 2 min, 10 ml of a soln. adjusted to pH 1·6 and containing 5 to 60 μg of Ti^{IV} and 2 ml of M-$NaClO_4$ with 5 ml of 2·5mM-reagent in $CHCl_3$. Re-extract the aq. phase with 4 ml of $CHCl_3$, combine the extracts, dilute them to 10 ml with $CHCl_3$, and measure the absorbance at 355 nm *vs.* a reagent blank. Phosphate, Fe^{III}, V^V and Mo^{VI} interfere, but the effect of Fe^{III} or V^V can be masked by ascorbic acid and that of Mo by mercaptoacetic acid. Many other common ions can be tolerated. W. C. D.

1B129. **Spectrophotometric determination of titanium as a mixed thiocyanate - 3-hydroxy-2-methyl-1-phenyl-4-pyridone complex.** Tamhina, B.; Vojkovic, V.; and Herak, M. J. (Lab. Anal. Chem., Fac. Sci., Univ., Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1977, **49** (3), 533–537.—An improved procedure for the determination of Ti is described; it involves extraction of Ti from 2M-HCl or M-H_2SO_4 into $CHCl_3$ as the Ti - SCN^- - reagent (1 : 2 : 2) complex. At this acidity there is no polymerisation of SCN^- and the $CHCl_3$ phase can be used for spectrophotometry at 365 nm without filtration. Beer's law is obeyed for 0·1 to 2·0 μg ml^{-1} of Ti. Niobium and Ta should be removed before determination of Ti; Fe and Cu, which interfere, can be masked by ascorbic acid, and Mo and W by mercaptoacetic acid; 22 other metals and 11 anions are tolerated. B. D. H.

1B130. **Simultaneous spectrophotometric determination of titanium(III) and titanium(IV) by using benzoylacetone.** Opasova, R. G.; Savostina, V. M.; and Lobanov, F. I. (M. V. Lomonosov Moscow State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 974–976 (in Russian).—Both Ti^{III} and Ti^{IV} form coloured complexes with benzoylacetone (**I**) in a medium of aq. 50% ethanol $\simeq 0{\cdot}05$M in HCl, 0·08M in **I** and containing 0·8% of $SnCl_2$ (to stabilise the Ti^{III} complex). The Ti^{III} and Ti^{IV} complexes exhibit absorption max. at 615 nm ($\epsilon = 3000$) and 382 nm ($\epsilon = 8500$), respectively. Beer's law is obeyed for 0·4 to 8 μg ml^{-1} of Ti, and Fe, Ni, Co, Cr, Al and lanthanoids do not interfere. The coeff. of variation was $<4{\cdot}5\%$ (5 results) when determining 5 to 50 μg of Ti in soln. containing Al, Cr, Co, Ni and Fe (100 μg of each). M. P.

1B131. **Extraction - spectrophotometric determination of titanium after separation from iron by a chloroform solution of 2-bromobutyric acid containing 3-methyl-1-phenylpyrazolin-5-one.** Pyatnitskii, I. V.; and Simonenko, V. I. (T. G. Shevchenko Kiev State Univ., USSR). *Ukr. Khim. Zh.* (*Russ. Ed.*), 1977, **43** (4), 416–417 (in Russian).—The test soln. is made M in HCl and Fe^{3+} are reduced to Fe^{2+} by powdered silver. The soln. is then analysed as described previously (*Anal. Abstr.*, 1977, **32**, 2B111). G. S. S.

1B132. **Application of atomic-absorption analysis to [titanium - nickel - aluminium] alloy.** Vinogradov, L. N.; Skomorokhov, B. A.; Rasokhina, T. P.; and Rakhmanov, N. S. *Tekhnol. Legk. Splavov. Nauch.-Tekh. Byul. VILSa*, 1977, (2), 64–65 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G204.

1B133. **Quantitative analysis of hydrogen in titanium with an ion micro-analyser.** Okajima, Yoshiaki; Aizawa, Yukiyoshi; Suzuki, Katsumi; and Sugawara, Yasushi (Res. Lab., Hitachi Ltd., Kuzimachi, Hitachi-shi, Ibarachi, Japan). *Bull. Chem. Soc. Jpn.*, 1977, **50** (4), 886–888.—Argon was ionised by a duoplasmatron and the resulting ions (beam diam. 1 mm) were accelerated at 10 kV on to the sample (5 mm × 5 mm; 1 mm thick), which was mounted with silver paste in a holder. The ions sputtered from the sample surface were accelerated at 3 kV and analysed by m.s. The ratio of the peaks at *m/e* 49 and 46 (*i.e.*, $^{48}TiH^+$ and $^{46}Ti^+$) was used to determine H^+ concn. A rectilinear relationship was obtained between this ratio and H concn. (0·001 to 0·5%) as determined by the vacuum fusion method. Depth concn. profiles for titanium treated by cathodic polarisation are shown. E. A.

1B134. **Determination of silicon in metallic titanium and some of its alloys.** Gorlova, M. N.; Feofanova, N. M.; and Kornyushkova, Yu. D. *Tekhnol. Legk. Splavov. Nauch.-Tekh. Byul. VILSa*, 1977, (2), 68–70 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G194.

1B135. **Improvements in the spectrofluorimetric determination of zirconium with bromopyrogallol red.** Kiskyte, M.; and Ramonaite, S. *Liet. TSR Aukst. Mokyklu Mokslo Darb., Chem. Chem. Technol.*, 1976, (18), 3–8 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G131.

1B136. **Spectrophotometric determination of zirconium(IV) with chlorsulphophenol S [3,6-bis-(5-chloro-2-hydroxy-3-sulphophenylazo)chromotropic**

acid]. Ishikura, Fumiko; and Tonosaki, Koichi (Fac. Sci., Hirosaki, Japan). *Sci. Rep. Hirosaki Univ.*, 1976, **23** (1), 24–28 (in Japanese).—In 0·6M-$HClO_4$ chlorsulphophenol S forms with Zr^{IV} a blue complex that can be used in determining micro amounts of Zr. The complex has an absorption max. at ≃645 nm in the presence of at least a twofold excess of reagent. Beer's law is obeyed for 4·2 to 42 μg of Zr^{IV} in 25 ml; the Sandell sensitivity is 2 ng cm^{-2}, and the ε value is ≃39,000. Gallium, Bi^{III}, Cu^{II}, Th and edta interfere seriously.
CHEM. ABSTR.

1B137. **Determination of low concentrations of zirconium in rocks with xylenol orange.** Dorosh, V. M.; Sukharenko, A. V.; and Fominykh, I. M. *Tr. Inst. Geol. Geofiz. Akad. Nauk SSR, Sib. Otd.*, 1977, (315), 95 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (16), Abstr. No. 16G183.

1B138. **Colour reactions of arsenazo III with zirconium and hafnium in various media.** Nemodruk, A. A.; and Boganova, A. N. (State Sci.-Res. and Design Inst. Alloys and Non-Ferrous Met. Process., Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1131–1137 (in Russian).—Arsenazo III forms 2:1 (ligand-to-metal) complexes with Zr^{IV} and Hf^{IV} in aq. 10 to 20% acetone media that are ≃8M in HCl and <50μM in the reagent (an 8-fold molar excess of which, relative to Zr or Hf, is needed); either complex is extractable into a higher alcohol (*e.g.*, hexanol) and exhibits max. absorption at 665 nm (ε ≃142,000). With the metal present in excess, 1:1 complexes are formed; these are not extractable into water-immiscible solvents, and exhibit max. absorption at 665 nm (ε ≃73,000). In media 7 to 9M in HNO_3 or $HClO_4$, the ε values of the 1:2 complexes are only ≃100,000. M. P.

Group V

N, P, As, Sb, Bi, V, Nb, Ta

1B139. **Determination of nitrogen monoxide in standard gases.** Rechenberg, W. (Forschungsinst. Zementind., Duesseldorf, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **285** (1), 48 (in German).—The NO is converted into NO_2^- by absorption in an alkaline KHF_2 soln. The NO_2^- are then used to diazotise sulphanilic acid, and, after coupling with 1-naphthylethylenediamine, the absorbance of the red - violet azo-dye formed is measured at 546 nm. The standard deviation for NO concn. from 250 to 800 p.p.m. is 4·5 p.p.m. and at 2100 p.p.m. it is 12 p.p.m. The method is free from bias. J. K. C.

1B140. **Effect of thiocyanate on nitrite estimation and cleavage of nitrosamines.** Boyland, E.; and Woolf, A. S. (Inst. Occup. Health, Sch. Hyg. and Trop. Med., London, W.C.1, England). *IARC Sci. Publ.*, No. 14, 1976. Pp. 161–171.—Both SCN^- and Br^- enhance the intensity of the colour formed during photometric determination of NO_2^- with sulphanilic acid and *N*-1-naphthylethylenediamine; SCN^- catalyse the diazotisation of sulphanilic acid and inhibit the reaction of NO_2^- with the naphthylethylenediamine. Although SCN^- catalyse the denitrosation of most *N*-nitrosamines only slightly they strongly catalyse that of methylnitrosoaniline. Methylnitrosoaniline is also denitrosated by sulphanilic acid. Nitrate is released from nitrosamines on heating with HBr in acetic acid during the determination of nitrosamines as described by Eisenbrand and Preussmann (*Anal. Abstr.*, 1971, **21**, 1469); SCN^- are less active than Br^- under the conditions used, and thus cannot be used for determining nitrosamines. CHEM. ABSTR.

1B141. **Fluorimetric method for determination of nitrate with 2-phenylbenzothiazole.** Nakano, Saburo; Yoshida, Tomohiko; Mikoshiba, Kazuo; and Taniguchi, Hirokazu (Meiji Coll. Pharm., Setagaya-ku, Tokyo, Japan). *Chem. Pharm. Bull.*, 1977, **25** (6), 1237–1242.—The determination of NO_3^- at levels of, *e.g.*, 0·01 to 3 μg ml^{-1} is based on reaction with 2-phenylbenzothiazole in conc. H_2SO_4 medium; the resulting 6-nitro-2-phenylbenzothiazole is extracted into ethyl ether, the extract is evaporated, and a soln. of the residue in ammoniacal dioxan is submitted to fluorimetry at 457 nm, with excitation at 358 nm. Interference from Cl^-, ClO^-, NO_2^- or Fe^{2+} could be prevented by the presence of sulphamic acid, Na_2SO_3 or Ag_2SO_4. The procedure has been applied to tap-water. F. R. B.

1B142. **Collector sensitivity: source of error in nitrogen-15 isotopic analysis.** Kerven, G. L.; and Saffigna, P. G. (Dept. Agric., Univ. Queensland, St. Lucia, Australia). *Int. J. Mass Spectrom. Ion Phys.*, 1977, **23** (4), 333–336.—Measurements of enrichment in ^{15}N indicated that a change in collector sensitivity in the Nier-type double-collector system could occur within 1 week and could cause an error in absolute isotopic content and in enrichment value. This change in sensitivity was due to broadening of the ion-beam images formed at the collector plates, causing some of the $^{14}N^{15}N$ to be measured as $^{14}N_2$. The beam broadening is attributed to a change in electron optics caused by bending of the rhenium filament. An equation is presented for calculating the relative collector sensitivity and the corrected ratio of $^{14}N^{15}N$ to $^{14}N_2$. The validity of the correction was tested during a 7-month measurement of ^{15}N enrichment of ^{15}N-labelled ammonium sulphate. Use of tungsten as filament material instead of rhenium eliminated bending of the filament, but led to a higher background at $m/e = 28$ (due to formation of CO). CHEM. ABSTR.

1B143. **Activation transverse cross-sections of nuclear reactions leading to the radio-isotope oxygen-14.** Muminov, V. A.; Mukhammedov, S.; Sultanov, B.; and Rakhmanov, A. (Inst. Yad. Fiz., Ulugbek, USSR). *Dokl. Akad. Nauk Uzb. SSR*, 1976, (3), 26–27 (in Russian).—To develop activation methods for determining N and C, the activation cross-section of the reactions $^{14}N(p,n)^{14}O$ and $^{12}C(^3He,n)^{14}O$ was measured. Cellulose nitrate and polystyrene samples were irradiated with up to 18-MeV protons and up to 24-MeV ^3He nuclei, respectively, at 0·01 μA for 1 min. After a 1 to 2-min decay period, the γ-activity of ^{14}O was measured at 2·31 MeV with a Ge(Li) detector and a multi-channel analyser. The relative errors were <20%. CHEM. ABSTR.

1B144. **Systematic analysis of nitrogen compounds in an aqueous solution.** Takahashi, Kazumasa; Fuse, Shinzo; and Mogi, Junichi (Ishikawajima-Harima Heavy Industry Co. Ltd., Tokyo, Japan). *Ishikawajima-Harima Giho*, 1976, **16** (3), 246–252 (in Japanese).—A method is described for the determination of nitrates, nitrites, ammonium salts and sulphamates in scrubbing soln. used for flue-gas treatments. Sulphamates were converted into NH_4^+ salts by heating in acid soln., nitrates and nitrites were reduced to NH_4^+ by heating with

Devarda's alloy in alkaline soln., and nitrites were reduced to N by sulphamic acid; the products were then determined after steam-distillation of the pre-treated soln. The errors were 7, 2, 5, and 1%, respectively, for the determination (in mg l^{-1}) of NO_2^- (60), NO_3^- (50), NH_4^+ (13), and sulphamate (40), respectively. CHEM. ABSTR.

1B145. **Pre-column synthesis of trimethylsilyl derivatives of aqueous phosphate for gas-chromatographic analysis.** Getty, Richard H.; Stone, Julia; and Hanson, Richard H. (Chem. Dept., Univ. Arkansas, Little Rock, Ark. 72204, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1086–1088.—A 5-μl portion of aq. PO_4^{3-} (0·25 to 5 μg) is placed in a reaction tube plugged with silanised glass wool, H_2O is removed by heating at 98° under reduced pressure, and to the cooled contents of the tube is added 80 μl of *NO*-bis(trimethylsilyl)trifluoroacetamide - chlorotrimethylsilane (99 : 1). After heating at 80° for 15 min, the tube is heated to 140° during 5 min, then the system is flushed by passage of N (at 60 ml min^{-1}), the excess of silylating reagent and some by-products being vented off. The trimethylsilyl derivative is heated at 45° and swept on to the glass g.l.c. column (6 ft. × 0·25 in.), which is packed with 5% of OV-225 on silanised AW Chromosorb W (60 to 80 mesh) and operated at 250°, with dual flame ionisation detection. The method can also be applied to those organic acids that are non-volatile at temp. <100°. R. W.

1B146. **Spectrophotometric determination of phosphorus as an ion-association compound between molybdophosphoric acid and butylrhodamine B [the butyl ester of C. I. Basic Violet 10].** Demina, L. A.; Mazepova, N. N.; Zhdanov, S. I.; and Podol'skaya, B. L. (All-Union Sci.-Res. Inst. Chem. Reagents and Special-Purity Chemicals, Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1138–1141 (in Russian).—Molybdophosphoric acid (**I**) forms a 1 : 1 complex with butylrhodamine B (**II**) in an aq. medium 0·6M in HNO_3, 2mM in $(NH_4)_6Mo_7O_{24}$ and 0·4mM in **II**. This complex exhibits max. absorption at ≃560 nm; the best analytical wavelength is 580 nm (ϵ = 45,000). To determine 0·2 to 0·4 p.p.m. of P (as PO_4^{3-}) in ammonium chloride (0·5 g), the sample is dissolved in H_2O (5 ml), aq. 5% $(NH_4)_6Mo_7O_{24}.4H_2O$ (0·3 ml) and 9M-HCl (0·4 ml) are added and the **I** formed is extracted into butanol - $CHCl_3$ (3 : 7) (4 ml) and re-extracted into aq. 0·2M-NaOH (2 ml). This aq. soln. is neutralised with HCl; 7M-HNO_3 (0·4 ml), 5% $(NH_4)_6Mo_7O_{24}.4H_2O$ soln. (0·3 ml) and aq. 0·02% **II** (1 ml) are added, and the absorbance is measured at 580 nm. The coeff. of variation is <7·5% (9 results). The method can be also used for determining traces of PO_4^{3-} in hydrochloric and hydrobromic acids. Any As present must be removed by first evaporating the test soln. with KI. M. P.

1B147. **Determination of trace impurities in high-purity phosphorus by atomic-absorption spectrophotometry.** Wunderlich, E.; and Haedeler, W. (Zentrallab., Preussag A.-G., Goslar, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 19–21 (in German).—For the determination of Pb, Cu, Zn, Cd, Mn, Ca, Mg, Na and K, a 1-g sample of red phosphorus is treated with conc. HNO_3 (15 ml) and then, with evaporation, with HCl (1 : 1) (5 ml). The mixture is then evaporated with a further 5 ml of conc. HNO_3 and the HNO_3 remaining is destroyed by evaporation with 20% formic acid (5 ml). The residue is taken up in H_2O (120 ml), and the soln. is passed through a silica column (6 cm × 1 cm) packed with Dowex 50W-X8 resin (50 to 100 mesh; H^+ form) at 3 to 4 ml min^{-1}, followed by H_2O (100 ml). The cations are eluted with HCl (1 : 1) (40 ml) at 2 to 3 ml min^{-1} followed by H_2O (20 ml), the eluate is evaporated to dryness, the residue is dissolved in HCl (1 : 1) (1 ml) and H_2O (5 ml), and the soln. is diluted to 10 ml and examined by a.a.s. or, for alkali metals, by flame a.e.s. For the determination of Fe and Al, a 1-g sample dissolved in conc. HNO_3 is evaporated with HCl and $HClO_4$, and after addition of H_2O (≃90 ml) and aq. 25% ammonium tartrate (1 ml) the soln. is brought to pH 10 with aq. NH_3 and extracted with six 10-ml portions of a soln. of 1 g of 8-hydroxyquinoline (**I**) and 2 ml of butylamine in 100 ml of $CHCl_3$. The combined extracts are evaporated, the **I** is expelled by heating, the residue is taken up in 60% $HClO_4$, the soln. is evaporated to dryness and the residue is taken up in HCl (1 : 1) - H_2O for a.a.s. For the determination of As, a 2-g sample dissolved in conc. HNO_3 is evaporated with HCl and $HClO_4$, and after dissolving the residue in H_2O (3 ml) and addition of 48% HBr (45 ml) the soln. is extracted twice with benzene (25 ml). The extract is washed with 8M-HBr (2 × 5 ml), and the As is extracted into H_2O. From the aq. phase AsH_3 is generated and absorbed in 10 ml of a soln. of 625 mg of Ag diethyldithiocarbamate and 410 mg of ephedrine in 250 ml of $CHCl_3$, in which the As is determined spectrophotometrically at 546 nm with use of a 4-cm cell. For 10 to 20 p.p.m. of impurities the coeff. of variation ranged from 3% (for Cd) to 13% (for Na, Ca and Fe). H. L. L.

1B148. **Azine dyes as new indicators in the bromatimetric determination of arsenic(III).** Rao, N. Venkateswara; and Ramana, P. V. (Chem. Dept., Andhra Univ., Waltair, India). *Fresenius' Z. Anal. Chem.*, 1977, **285** (2), 125.—Six azine dyes [Azocarmine B (C.I. Acid Red 103), Azocarmine G (C.I. Acid Red 101), Wool fast blue GL (C.I. Acid Blue 102), neutral red (C.I. Basic Red 5), neutral violet (C.I. 50030) and nigrosine (C.I. Acid Black 2)] are suitable indicators for direct titration of As^{III} with $KBrO_3$ in the absence of added Br^- and in 0·5 to 1·0M-HCl or 2 to 2·5M-H_2SO_4. The first three dyes can be used in 0·01M acid in the presence of OsO_4. They may also be used in the reverse titration in neutral soln. or in 0·05 to 0·25M-H_2SO_4 with OsO_4 present. Of the six dyes, Azocarmine G is preferred. D. B. A.

1B149. **Pre-concentration and voltammetric determination of arsenic by using a stationary mercury micro-electrode.** Toropova, V. F.; Polyakov, Yu. N.; and Soboleva, L. N. (V. I. Ul'yanov-Lenin Kazan State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 985–988 (in Russian).—Arsenic can be concentrated on such an electrode from a soln. 2 to 20μM in As^{III} and 6M in HCl; the electrode is kept in the test soln. (at 40°) for 10 min without applying an external potential, then transferred to M-KOH for scanning the voltammetric curve of the oxidation of the deposited As from −0·5 to −0·1 V *vs.* a silver - AgCl electrode. The height of the oxidation-current peak at ≃ −0·32 V is rectilinearly related to the concn. of As^{III} in the test soln. There is no interference from up to 100-fold amounts (relative to As) of Bi, Sb^{III}, Sn^{II} or Pb^{II} or up to a 10-fold amount of Cu^{II}, but Fe^{III} and strong oxidants interfere. When the method was used to determine 4·5 to 7·5 μg ml^{-1} of As^{III} in prepared soln. 0·5mM in Bi, Sb, Pb and Sn and 0·1mM in Cu, the coeff. of variation was <4·1% (3 results). M. P.

1B150. **High-precision gravimetric 'coulometry' using the silver - perchloric acid coulometer: titration of arsenous oxide with electro-generated iodine.** Newton, C. M. (Bell Laboratories, Murray Hill, N. J. 07974, U.S.A.). *Talanta*, 1977, **24** (6), 377–381.—This technique is evaluated as an alternative to conventional titrimetry. The loss of wt. by electrolytic dissolution of a highly-pure silver anode in series with the cathode of a conventional constant-current titration system is measured and related to the number of equiv. of substance titrated. The precision of the method is determined by titrations of the NBS Standard Reference Material 83C arsenous oxide with electro-generated iodine, with amperometric end-point detection by means of two polarised electrodes. Depending on the size of the sample, ultimate precision of 25 p.p.m. is obtained. T. J. S.

1B151. **Determination of arsenic and antimony in geological materials by flameless atomic-absorption spectrometry.** Aslin, G. E. M. (Geol. Surv. Canada, Ottawa, Ont.). *J. Geochem. Explor.*, 1976, **6** (3), 321–330.—Samples (250 mg) were decomposed by aqua regia or by evaporation with HF then HNO_3 - $HClO_4$, the residue being dissolved in HCl. After further dilution with dil. HCl, aliquots were injected into the sample cell, which contained $NaBH_4$ soln. A stream of N (2 l min^{-1}) swept the resulting hydrides into a heated silica tube; As was measured at 193·7 nm and Sb at 217·6 nm. A second stream of N flowed across the ends of the tube to prevent combustion of H (formed in the reduction process) in the flame heating the tube. Sixteen standard reference materials (As 0·4 to 430 mg kg^{-1}; Sb 0·2 to 115 mg kg^{-1}) were taken through both sample-preparation methods, and gave calibration graphs identical to those given by standard soln. (in 10% aqua regia). For soln. containing up to 50 ng ml^{-1} of As or Sb there was no interference from up to 5 μg ml^{-1} of Fe, Co or Cu; Ni interfered at the 1 or 5 μg ml^{-1} level, but could be masked by edta, as could Ag (1 μg ml^{-1}). The detection limits (based on a sample wt. of 500 mg) were 0·08 μg g^{-1} for Sb and 0·16 μg g^{-1} for As. J. P. A.

1B152. **Spectrophotometric study of triarylmethane, azine, oxazine and thiazine dyes and their reactions with antimony in aqueous solution. IV. Janus green B and its ionic associate with antimony(V).** Naruskevicius, L.; Kazlauskas, R.; and Bumialis, V. *Liet. TSR Aukst. Mokyklu Mokslo Darb., Chem. Chem. Technol.*, 1976, (18), 21–24 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G156.

1B153. **Stripping-voltammetric determination of antimony in presence of tellurium.** Neiman, E. Ya.; and Sumenkova, M. F. (State Sci.-Res. and Design Inst. Alloys and Non-ferrous Metals Process., Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 989–994 (in Russian).—The method involves deposition of Sb and Te on a mercury-plated-graphite electrode (by electrolysis at −0·5 V *vs.* the s.c.e. for 2 to 5 min) from a medium M in HCl and containing 5 μg ml^{-1} of Hg^{II}. The magnitude of the subsequent oxidation current is rectilinearly related to Sb concn. in the range 2 to 250 ng ml^{-1} and is independent of the concn. of Te^{IV} provided that the molar ratio of Sb to Te is <5. The method has been used for determining ≃1·85 p.p.m. of Sb in a zinc alloy containing 10 p.p.m. of Te. The coeff. of variation was 15% (8 results), and the results agreed with those of spectrophotometry and spectrography. M. P.

1B154. **Reaction of alizarin green with bismuth(III).** Simek, Jiri; and Ruzicka, Eduard (Fac. Nat. Sci., Palacky Univ., Olomouc, Czechoslovakia). *Acta Univ. Palacki. Olomuc., Fac. Rerum Nat.*, 1976, **49** (Chem. 15), 183–189 (in German).—The reaction between alizarin green (H*L*) and Bi^{III} in acid media has been studied spectrophotometrically. Spectrophotometric titration of Bi with H*L* suggested that two complexes (Bi*L* and BiL_2) were formed. H*L* is recommended as indicator for determining mg amounts of Bi by complexometric titration with edta. CHEM. ABSTR.

1B155. **Spectrophotometric determination of bismuth(III) with chlorsulphophenol S.** Ishikura, Fumiko; and Tonosaki, Koichi (Fac. Sci., Hirosaki Univ., Japan). *Sci. Rep. Hirosaki Univ.*, 1976, **23** (1), 29–33 (in Japanese).—Chlorsulphophenol S reacts with Bi^{III} to form a 1 : 1 blue complex having an absorption max. at 640 nm (ϵ ≃27,000). The absorbance remains constant over the pH range 4·2 to 5·1; Beer's law is obeyed for 10·5 to 83·6 μg of Bi^{III} in 25 ml at pH 4·7, and the Sandell sensitivity is 8 ng cm^{-2}. Aluminium, Cu^{II}, Ga, Th, V^{IV}, Y and edta interfere seriously. CHEM. ABSTR.

1B156. **Thiocyanate complexes of bismuth with pyrazolone derivatives: photometric determination of bismuth.** Gvelesiani, L. T.; Torondzhadze, D. D.; and Akimov, V. K. (Tbilisi State Univ., USSR). *Soobshch. Akad. Nauk Gruz. SSR*, 1976, **84** (3), 597–600 (in Russian).—Bismuth was extracted into $CHCl_3$ as the Bi - 1,1-diantipyrinylbutane - SCN^- (1 : 1 : 4) ion-association complex from 0·07N-H_2SO_4 medium; after 15 to 20 min, the absorbance of the extract was measured at 330 nm (ϵ ≃14,000). Beer's law was obeyed for 4·0 to 10·0 μg ml^{-1} of Bi. The coeff. of variation was 2·74% when determining 4·0 μg of Bi; interference was caused by Fe, Ti, Cu and Sn. Data are also given for the extraction of Bi - SCN^- complexes with antipyrine, diantipyrinylmethane, 1,1-diantipyrinylpropane or αα-diantipyrinyltoluene into $CHCl_3$ and dichloroethane. CHEM. ABSTR.

1B157. **Thermal decomposition of analytically important thiocyanatobismuthates(III).** Cyganski, Andrzej (Inst. Gen. Chem., Tech. Univ., Lodz, Poland). *Talanta*, 1977, **24** (6), 361–366.—The thermal stabilities of $Cs[Bi(SCN)_4]$ and $Cs_3Na[Bi(SCN)_6]$, also $Rb[Bi(SCN)_4]$, have been studied, and the mechanism of their thermal decomposition has been evaluated by analysis of their sinters. Safe temp. for drying the compounds for gravimetric determinations were established, and the analytical potential of the thermal reactions is discussed. The procedure for analysis of the sinters could be applied to other salts of Bi. T. J. S.

1B158. **Complex of vanadium(V) with anthranilic acid benzohydrazide and its use in analysis.** Karpova, O. I.; Pilipenko, A. T.; and Lukachkina, V. V. (Inst. Colloid Chem. and Water Chem., Acad. Sci. Ukr. SSR, Kiev). *Zh. Anal. Khim.*, 1977, **32** (6), 1142–1145 (in Russian).—The named reagent (**I**) forms a 1 : 1 : 2 : 1 [VO^{3+} - **I** - HSO_3^- (or Cl^- or NO_3^-) - acetone] complex with V^V in aq. 60 to 80% acetone that is M to 4M in H_2SO_4 or 1·5 to 3·5M in HCl (or HNO_3). This complex exhibits max. absorption at 440 nm (ϵ = 5200) in the presence of H_2SO_4, and the colour is stable for 50 min; at least a 40-fold molar excess of **I** relative to V^V is needed. Beer's law is obeyed for 0·08 to 8 μg ml^{-1} of V^V; no interference is caused by V^{IV}, Cu^{II}, Ca, Nb^V, Ta^V, Mo^{VI}, W^{VI},

Os^{VIII}, Mn^{II}, Pd^{II}, Pd^{IV}, Pt^{IV} or small amounts of Mn^{VII}, and Fe^{III} can be masked by H_3PO_4, but strong oxidants (ClO_3^-, IO_3^-, BrO_3^-, $Cr_2O_7^{2-}$ and H_2O_2) interfere. A method based on these findings has been used for determining 0·01 to 10% of V in steel. The coeff. of variation was 2% (6 results) for 1·04% of V in a steel also containing 14% of W and 3·4% of Cr.
M. P.

1B159. **Equilibria in micellar systems. Photometric determination of vanadium with 6-hydroxy-5-oxodibenzo[*a*,*j*]phenoxazine-8,11-disulphonic acid activated by hexadecyltrimethylammonium bromide.** Lasovsky, J.; and Ruzicka, E. (Dept. Inorg. and Phys. Chem., Palacky Univ., Olomouc, Czechoslovakia). *Collect. Czech. Chem. Commun.*, 1977, **42** (6), 1941–1948.—A study is presented of complex formation between V^V and the cited compounds, and a procedure is described for determining 9 to 90 μg of V. To the test soln. are added 10 ml of mM dye, 2·5 ml of acetate buffer of pH 4·0 and 15 ml of 4mM-hexadecyltrimethylammonium bromide, the mixture is diluted to 50 ml with H_2O, and the absorbance at 630 nm is measured against H_2O. The Sandell sensitivity is 2·6 ng cm^{-2}. Tervalent Fe, Fe^{II}, Mo^{VI}, Th, Zr^{IV}, W^{VI}, Sb^{III}, U^{VI}, Ti^{IV}, Pd^{II}, Sn^{IV} and Cr^{VI} interfere.
J. M. B.

1B160. **Polarographic behaviour of vanadium(III) and vanadium(IV) in the presence of thiocyanate.** Grigor'eva, M. F.; Kasatochkina, L. A.; and Tserkovnitskaya, I. A. (A. A. Zhdanov Leningrad State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1146–1148 (in Russian).—Both V^{III} and V^{IV} are reduced to V^{II} at a dropping-mercury electrode in 0·5M-KSCN (0·1M in H_3PO_4) as the supporting electrolyte. The V^{IV} is reduced in two one-electron steps ($E_{\frac{1}{2}}$ −0·21 and −0·55 V *vs.* the s.c.e.); the latter wave corresponds to the reduction of V^{III}. The limiting reduction currents are rectilinearly related to the concn. of V^{III} and V^{IV}; there is no interference from <50-fold amounts of V^{IV} relative to V^{III}, and vice versa. A method based on these findings has been used for determining 0·33 to 18·8% of V^{III} and 1·8 to 49% of V^{IV} in Li - Zn - V spinels and in a phosphate glass also containing Zn, Ba and K; samples are dissolved in 4M-H_3PO_4 in an atmosphere of N. The V^{IV} is determined from the height of the wave at −0·21 V, and the sum of V^{IV} plus V^{III} is found from that at −0·55 V. The coeff. of variation were <18% (8 results) for V^{III} and <5% for V^{IV}, and the results agreed with those by spectrophotometry.
M. P.

1B161. **Use of atomic absorption for continuous analytical control of the refining of refractory metals.** Timofeev, E. F.; Lalayan, M. S.; Elyutin, A. V.; Maiorov, I. A.; and Nedler, V. V. *Zh. Prikl. Spektrosk.*, 1977, **26** (3), 397–403 (in Russian).—The electron-beam vacuum-fusion refining of vanadium containing <3·0% (w/w) of Fe can be monitored by a.a.s. of the Fe (at 302·06 nm) in the space above the metal crystalliser. The coeff. of variation were 0·34 to 0·37% and 0·05 to 0·09% for determining 0·005 to 0·36% and 0·5 to 3·0% of Fe, respectively. The rate of evaporation of Fe can be found by measuring the integral absorbance.
CHEM. ABSTR.

1B162. **Elementary analysis of vanadium and titanium phosphates by activation analysis with fast neutrons.** Vaivads, J.; Gedrovics, J.; Konstants, Z.; and Pelekis, L. (Inst. Neorg. Khim., Riga, Latv. SSR). *Latv. PSR Zinat. Akad. Vestis, Fiz. Teh. Zinat. Ser.*, 1976, (5), 8–14 (in Russian).—The samples (including a titanium phosphate glass) were analysed by non-destructive 14-MeV n.a.a. with use of a flux (monitored with copper foil) of ≃10^7 n cm^{-2} s^{-1} and γ-ray spectrometry with a Ge(Li) detector and a 1024-channel analyser. To determine V, P, Ti and O in a 0·5-g sample, the line intensities were measured at 320·1, 1778·8, 1312·1 and 3500 keV for ^{51}Ti, ^{28}Al, ^{48}Sc and ^{16}N, respectively; V_2O_5, TiO_2 and P_3N_5 were used as standards, and the irradiation times were 15 min, 5 min and 5 h for the determination of V, P and Ti, respectively. The results agreed well with those obtained by chemical methods, and relative errors did not exceed 4%.
CHEM. ABSTR.

1B163. **Analytical application of oleophilic quaternary ammonium salts. X. Extraction of the niobium - catechol violet - tridodecylethylammonium ternary complex.** Shijo, Yoshio (Dept. Ind. Chem., Fac. Eng., Univ., Ishii-machi, Utsunomiya, Japan). *Bull. Chem. Soc. Jpn.*, 1977, **50** (4), 1011–1012.—Niobium is extracted at pH 4·5, from an aq. medium (25 ml) 0·06mM in catechol violet (**I**), into CCl_4 (5 ml) containing mM-tridodecylethylammonium bromide (**II**) by shaking for 5 min. The absorbance of the organic phase is measured at 553 nm; the calibration graph is rectilinear for 0·58 to 7 μg of Ni. Uranium, Th, Mo, W, Al, Fe, V, NO_3^-, I^-, SCN^- and ClO_4^- interfere, but interference from Cu, Bi and Sn can be masked by 2-mercaptoethanol. Results indicate that the molar ratio of Nb to **I** to **II** in the complex is 1:2:3. (*Cf. Anal. Abstr.*, 1977, **33**, 3B57.)
E. A.

1B164. **Determination of low concentrations of niobium and tantalum in rocks.** Dorosh, V. M.; Sukharenko, A. V.; and Fominykh, I. M. *Tr. Inst. Geol. Geofiz. Akad. Nauk SSSR, Sib. Otd.*, 1977, (315), 95–96 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G175.

1B165. **Determination of very small amounts of nitrogen and carbon monoxide in high-melting metals by hot-extraction levitation melting in ultra-high vacuum.** Winterkorn, M.; Schulze, K.; and Toelg, G. (Max-Planck Inst. Metallforsch., Stuttgart, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 27–39 (in German).—An apparatus is described for the r.f. levitation heating (at <0·1 μPa) of samples of niobium and the determination therein of evolved N and CO by high-resolution quadrupole m.s. The detection limits were 70 p.p.b. (N) and 50 p.p.b. (C plus O); the results are compared with those of other methods.
J. W. P.

Group VI

O, S, Se, Te, Po, Cr, Mo, W

1B166. **Analytical applications of solid-electrolyte probes: determination of oxygen in multi-component metal melts.** Sundermann, H.; and Schauer, V. (Int. At. Energy Ag., Nucl. Res. Centre, Karlsruhe, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 575–596 (in German).—Temperature - e.m.f. - concn. nomograms are presented for O in sodium, potassium, copper, nickel, cobalt and iron melts obtained by use of different solid electrolytes. An electronic computing system (shown in a diagram) is described whereby O concn. can be directly determined in multi-component alloys. A coulometer is used for calibration.
J. W. P.

1B167. **Effect of acidity of solutions on formation of methylene blue in the spectrophotometric determination of sulphur.** Krichevskaya, A. M.; and Fedorov, A. A. (I. P. Bardin Central Sci.-Res. Inst. Ferrous Metall., Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1149–1153 (in Russian).—Optimum conditions for determining S^{2-} as methylene blue (*cf.* Hofmann and Hamm, *Anal. Abstr.*, 1969, **16**, 119) have been established: the reaction mixture should be 0·63M in HCl, mM in *NN*-dimethyl-*p*-phenylenediamine and 1·85mM in Fe^{III}. The method has been used for determining S as impurity in hafnium. The metal sample (0·2 to 2 g) was dissolved at 300° in 30 to 70 ml of a mixture prepared by dissolving 1·5 g of H_3BO_3 and 1·5 g of KHF_2 in 300 ml of conc. H_3PO_4; the H_2S evolved was collected in 0·25M-NaOH (10 ml), and S^{2-} were converted into methylene blue under the stated conditions. The absorbance was measured at 668 nm; Beer's law was obeyed for 0·02 to 0·6 μg ml^{-1} of S, and the limit of determination was 1·2 p.p.m. of S in the sample. The coeff. of variation was $<8·7\%$ (10 results) when determining $\simeq 15$ p.p.m. of S. M. P.

1B168. **Potentiometric determination of sulphate with edta and the cupric-selective electrode.** Baumann, E. W. [Du Pont de Nemours (E.I.) and Co., Savannah River Lab., Aiken, S.C., U.S.A.]. *U.S. Energy Res. Dev. Adm., Rep.*, DP-1142, 1976. Pp. 12.—The SO_4^{2-} were pptd. as $BaSO_4$, which was dissolved in excess of ammoniacal edta, and the unconsumed edta was titrated potentiometrically with La^{3+}, a Cu^{2+}-selective electrode being used to detect the end-point. The method was used to determine mM concn. of SO_4^{2-} in soln. of metal oxides (in 3M-HCl), with a mean coeff. of variation of 3·5%. ERDA Energy Res. Abstr.

1B169. **Ultra-violet spectrophotometric determination of sulphite with hydrobromic acid.** Utsumi, Satori; Okutani, Tadao; and Sato, Yoshito (Coll. Sci. and Technol., Nihon Univ., Chiyoda-ku, Tokyo, Japan). *Bunseki Kagaku*, 1977, **26** (6), 383–386 (in Japanese).—It was found that soln. of SO_3^{2-} show an intense absorption max. at 285 or 300 nm in the presence of HCl or HBr, respectively, the absorbance being higher with HBr. To determine SO_3^{2-}, the soln. is mixed with 10 ml of 7·5N-HBr and diluted to 25 ml, and the absorbance is measured at 300 nm against H_2O. The coeff. of variation was 0·5% in the determination of 0·2mM-SO_3^{2-} (5 results). Tervalent Fe, Hg^{II}, Pb^{II}, NO_2^- and VO_3^- interfere.

1B170. **Determination of sodium thiosulphate with *N*-bromosuccinimide using Bordeaux red [C.I. Acid Red 17] as indicator.** (CSIR Lab., Lahore, Pakistan). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 43.—A dil. soln. of *N*-bromosuccinimide (**I**) is stable for one week if kept refrigerated and protected from light. The titration of 1 ml of dil. $Na_2S_2O_3$ soln. is carried out in the presence of 2 ml of 0·5% Na_2CO_3 soln. and 2 drops of a 0·05% aq. soln. of the indicator; 1 mol of $Na_2S_2O_3$ reacts with 4 mol of **I**. H. L. L.

1B171. **Simultaneous spectrophotometric determination of quadrivalent and sexavalent selenium.** Desai, G. R.; and Paul, J. (Chem. Dept., Univ. Bridgeport, Conn. 06602, U.S.A.). *Microchem. J.*, 1977, **22** (2), 176–181.—The method consists in formation (in the presence of ClO_4^-) and extraction of the Se^{IV} - diethyldithiocarbamate complex into $CHCl_3$ for measurements at 340 nm, followed by reduction of Se^{VI} (in the residual aq. phase) with 25% HBr and determination of the resulting Se^{IV} with diethyldithiocarbamate or (preferably) 3,3′-diaminobenzidine (**I**), each measurement being made at 340 nm. The calibration graphs are rectilinear for up to 120 μg ml^{-1} of Na_2SeO_3 or K_2SeO_4, and for up to 25 μg ml^{-1} of K_2SeO_4 when **I** is used. Accuracy and reproducibility over these ranges are high. W. J. B.

1B172. **Catalytic polarographic wave of bromate in presence of selenium(IV).** Toropova, V. F.; and Kopylova, O. V. (V. I. Ul'yanov-Lenin Kazan State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1159–1161 (in Russian).—In the reduction of BrO_3^- at a dropping-mercury electrode, a catalytic wave occurs at −0·9 V (*vs.* the s.c.e.) if Se^{IV} is also present. In the optimum supporting electrolyte, *viz*, formate buffer soln. of pH 3·6 that is 12mM in BrO_3^-, the catalytic current is rectilinearly related to the concn. of SeO_3^{2-} in the range 6 to 100μM; Te^{IV} interferes, but can first be oxidised to Te^{VI} by using $Cr_2O_7^{2-}$. The coeff. of variation was $<8·3\%$ (5 results) when determining 6·31 μg ml^{-1} of Se^{IV} in a prepared soln. also containing 10·2 μg ml^{-1} of Te^{VI}. M. P.

1B173. **Post-irradiation chemical separation of selenium in neutron-activation analysis.** Omi, Jun'ichi; and Hashimoto, Yoshikazu (Dept. Chem., Keio Univ., Kohoku-ku, Yokohama-shi, Kanagawa, Japan). *Bunseki Kagaku*, 1977, **26** (6), 419–422 (in Japanese).—Distillation of halides of Se, from a soln. (110 ml) containing 30 ml of each of 12N-HCl and 8·6N-HBr, separated Se from radioactive nuclides, except those of Hg and Sb, and from compounds which would interfere in subsequent solvent extraction. Recovery of Se in the distillation was 99·6 ± 0·3%. After concentration of the distillate, Se was extracted into benzene from an aq. medium of 12N-HCl - 60% $HClO_4$ (15 ml of each) and then re-extracted with aq. NH_3. Complete separation was achieved. The presence of HNO_3 lowered the extraction efficiency, but 97% recovery was attained if the HNO_3 concn. was $<3\%$. Over-all recovery was 97·2 ± 1·2% as found by a radioactive-tracer technique. Results for Se in bovine liver and a standard rock sample were in agreement with certified values.

1B174. **Determination of selenium in tellurium dioxide by neutron-activation analysis.** Johansen, O.; and Steinnes, E. (Inst. Atomenergi, Isot. Lab., Kjeller, Norway). *Int. J. Appl. Radiat. Isot.*, 1977, **28** (6), 599–600.—The sample ($\simeq$200 mg) was irradiated for 20 min in a thermal-neutron flux of $\simeq 1·5 \times 10^{13}$ n cm^{-2} s^{-1}. A mixture of irradiated sample, Se carrier and conc. HNO_3 was boiled, and conc. HCl was added. The soln. was then evaporated to dryness, a soln. of the residue in 2M-HCl was applied to a column of Dowex 1-X8 resin (Cl^- form), and the column was washed with 2M-HCl. To the combined percolate and washings were added conc. HCl, Te hold-back carrier and $Na_2S_2O_5$, and the mixture was heated. The pptd. elementary Se was collected on a membrane filter, and the γ-activity at 103 keV (due to ^{81m}Se) was counted with a Ge(Li) or NaI(Tl) detector. For standardisation, 0·5 ml of a soln. in 0·1N-HNO_3 containing 500 μg ml^{-1} of Se was irradiated, and 50 μl of the product was absorbed on filter-paper for activity measurement. The chemical yield ranged from 70 to 90%, and the limit of detection was $\simeq$0·1 p.p.m. W. C. D.

1B175. **Application of modern instrumental methods to trace analysis of high-melting metals.** Ortner, H. M.; and Lassner, E. (Metallwerk Plansee

A.-G., Reutte, Austria). *Mikrochim. Acta*, 1977, Suppl. VII, 41–62 (in German).—The suitability of different methods for the determination of heterogeneous and grain-boundary contaminants in sintered molybdenum, tungsten, niobium and tantalum is discussed, and new developments in X-ray fluorescence analysis and a.a.s. are mentioned in connection with these studies. Flameless a.a.s. and the use of Na_2WO_4-impregnated graphite tubes result in better precision and sensitivity for the determination of As, Pb and Si (50 to 500 p.p.m.) in molybdenum and tungsten. J. W. P.

1B176. **Determination of composition of mixtures of sodium chromite and chromic oxide by electron spin resonance spectrometry.** Goldberg, Ira B.; Crowe, Harry R.; and Robertson, Wayne M. (Sci. Center, Rockwell International, Thousand Oaks, Calif. 91360, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 962–966.—In an investigation of the properties of $NaCrO_2$ films formed on Cr-containing alloys in the presence of liquid sodium (used as a coolant in nuclear reactors), a method was developed for determining the Cr_2O_3 content of $NaCrO_2$ - Cr_2O_3 mixtures. The transition temp. from the antiferromagnetic to the paramagnetic form is lower for $NaCrO_2$ than for Cr_2O_3, and this fact permits separation of their e.s.r. signals. On the basis of relative signal amplitudes, determination of 0·2 to 10% of Cr_2O_3 in such mixtures can be effected, while relative double integrals can be used to determine 0·5 to 100% of Cr_2O_3. R. W.

1B177. **Spectrophotometric determination of tungsten as a mixed thiocyanate - 3-hydroxy-2-methyl-1-phenyl-4-pyridone complex.** Tamhina, B.; and Herak, M. J. (Lab. Anal. Chem., Fac. Sci., Univ. Zagreb, Yugoslavia). *Microchem. J.*, 1977, **22** (2), 144–148.—The W^{VI} is first reduced to W^V with $SnCl_2$ in HCl medium; the W^V - SCN^- complex is then formed and extracted into a soln. of the pyridone reagent (**I**) in $CHCl_3$ for spectrophotometry of the ternary complex at 406 nm. Optimum operating concn. are 5·2M-HCl, aq. 1·2% $SnCl_2$, aq. 1·5% KSCN and 7mM-**I**. The calibration graph is rectilinear for 2 to 30 μg ml^{-1} of W, and the Sandell sensitivity is 17 ng cm^{-2}. Most other metals (except U, Zr, Th, Mo^{VI}, Ti, Nb and Ta) could be tolerated in up to $\simeq$1000-fold excess (relative to W). W. J. B.

1B178. **Evaluation of photometric and gravimetric determinations of tungsten in minerals.** Kroshkina, A. B.; Shilova, L. K.; and Antonova, E. A. (All-Union Inst. Minerals, Moscow, USSR). *Zavod. Lab.*, 1977, **43** (6), 648–649 (in Russian).—The W was determined photometrically with use of an alkali-metal thiocyanate in the presence of a reducing agent, and gravimetrically by pptn. as tungstic acid from HNO_3 soln. and ignition to WO_3 at 700° to 750°. The photometric method is suitable for the determination of 0·01 to 5 mg of W (*i.e.*, up to 5% in a 0·1-g sample), whereas the gravimetric method is suitable for contents >2·5%. Both methods have good reproducibility. A. S. W.

1B179. **Analysis of tungsten bronzes.** Vondrak, Jiri (Inst. Inorg. Chem., Acad. Sci., Prague, Czechoslovakia). *Chem. Listy*, 1977, **71** (6), 561–570 (in Czech).—A review is presented, with 66 references. Special attention is given to sample preparation. H. M. T.

Group VII

F, Cl, Br, I, At, Mn, Tc, Re

1B180. **Determination of halide ions by cathodic-stripping analysis.** Manandhar, Krishna; and Pletcher, Derek (Dept. Chem., Univ. Southampton, England). *Talanta*, 1977, **24** (6), 387–390.—Conditions for cathodic-stripping analysis of halides at a mercury-drop electrode were studied. A rectilinear response was obtained over the concn. range 20μM to mM for soln. containing a single halide. Methods for analysing mixtures of halides are discussed. There were fewer interferences than for halide-selective electrodes; ClO_4^-, NO_3^-, PO_4^{3-} and SO_4^{2-} did not interfere at 0·1M, OH^- interfered at high concn., and S^{2-} caused serious interference even at 0·1mM. T. J. S.

1B181. **Determination of fluoride by photometric titration.** Moro Garcia, R.; Sanchez Uria, J. E.; and Arribas Jimeno, S. (Dept. Quim. Anal., Fac. Cienc., Univ. Oviedo, Spain). *Quim. Anal.*, 1977, **31** (1), 23–25 (in Spanish).—It is shown that F^- can be titrated with $TiCl_3$ soln., the end-point being determined by spectrophotometry of the green TiF_2^+ complex. A soln. (25 ml) of 0·05N-$TiCl_3$ in HCl or glycerol is diluted to 90 ml with water (acidified with HCl) and titrated with the test soln. ($\simeq$ 0·5N in F^-), with measurement at 450 nm. Use of the glycerol medium gave slightly lower errors. L. A. O'N.

1B182. **Thin-layer chromatography of groups of anions. I.** Gallego Andreu, R.; Bernal, J. L.; and Martinez, A. (Dept. Quim. Anal., Univ. Valladolid, Spain). *Quim. Anal.*, 1977, **31** (1), 3–7 (in Spanish).—The two groups Cl^-, Br^- and I^-, and ClO_3^-, BrO_3^- and IO_3^-, may be separated by ascending development on microcrystalline cellulose incorporating a fluorescent indicator. A suitable locating agent is aq. 1% $AgNO_3$ and, when u.v. illumination is necessary, a lamp providing 254- or 364-nm radiation can be used. Various mobile phases have been examined, *e.g.*, aq. acetone or aq. ethanol mixtures; addition of tartaric acid often improved the separation. The effects of NH_3 or organic bases in the mobile phase and of the concn. of anions have been examined. L. A. O'N.

1B183. **Solubilities of tetraphenylphosphonium and tetraphenylarsonium perchlorate and gravimetric perchlorate determination.** Siroki, M.; and Cesar, V. (Lab. Anal. Chem., Fac. Natural Sci. and Math., Univ., Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1977, **49** (3), 408–414.—The solubility of these two compounds in water and in aq. soln. of NaCl and of Na_2SO_4 was determined at temp. between 5° and 100° as a basis for the gravimetric determination of micro and semi-micro amounts of ClO_4^-. A recommended procedure for the determination of ClO_4^- with tetraphenylphosphonium chloride is given. The effect of salt concn. and the presence of several foreign anions on the accuracy and reproducibility of the results was investigated; I^-, MoO_4^{2-}, MnO_4^- and WO_4^{2-} interfered. B. D. H.

1B184. **Lower limits of the potentiometric titration of perchlorate using a perchlorate-ion-selective electrode.** Selig, Walter (Lawrence Livermore Lab., Univ. California, Livermore, Calif. 94550, U.S.A.). *Microchem. J.*, 1977, **22** (1), 1–6.—Solutions ($\simeq$0·05N) of tetraphenylarsonium chloride (**I**),

tetraphenylphosphonium chloride (**II**) and tetrapentylammonium bromide (**III**) were used as titrants for the determination of ClO_4^- with use of an Orion 93–81 ion-selective electrode and a double-junction reference electrode; the system was controlled by a PDP-8/I mini-computer. **III** was found to be unsuitable as titrant; however, down to 1·7mN-ClO_4^- could be determined with **I** or **II** and this limit could be lowered to 0·2mN if Gran plots were used. T. G. A.

1B185. **Solvent-extraction equilibria of acids. V. Extraction of hydrobromic, hydriodic and thiocyanic acids with trioctylphosphine oxide in hexane.** Niitsu, Masaru; and Sekine, Tatsuya (Fac. Pharm. Sci., Josai Univ., Sakado, Saitama, Japan). *Bull. Chem. Soc. Jpn.*, 1977, **50** (4), 1015–1016.—The extraction of these acids from M soln. of electrolytes (*viz*, H^+, Na^+ and ClO_4^-) into trioctylphosphine oxide soln. in hexane at 25° has been studied. Extraction constants are given. E. A.

1B186. **Pre-concentration of elements on impregnated paper with subsequent determination by 'peak' paper chromatography.** Aleskovskaya, V. N.; Aleskovskii, V. B.; and Bogdanova, E. G. (Inst. Textile and Light Ind., Leningrad, USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 922–925 (in Russian).—It is shown that I^- can be concentrated from soln. (containing $>2·5$ mg l^{-1} of I^-) on paper impregnated with AgCl. Up to 50 portions (each of 5 μl) of test soln. are applied to the same spot (the amount of I^- in the spot must not exceed 1 μg), and the I^- are then determined by 'peak' paper chromatography (*cf. Anal. Abstr.*, 1971, **20**, 1657). Similarly, Ru^{III} (>10 ng ml^{-1}) can be concentrated and determined on paper impregnated with metallic silver. M. P.

1B187. **Spectrophotometric study of the leuco-bases of malachite green [C.I. Basic Green 4] and Brilliant green [C.I. Basic Green 1]. [Determination of tri-iodide and sulphide.]** Ramanauskas, E.; and Jurkeviciute, J. *Liet. TSR Aukst. Mokyklu Mokslo Darb., Chem. Chem. Technol.*, 1976, (18), 50–55 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G16.

1B188. **Determination of water in iodine by infrared spectroscopy.** Farrow, R. N. P.; and Hill, A. G. (BDH Chemicals Ltd., Poole, Dorset, England). *Analyst* (*London*), 1977, **102** (1215), 480–481.—Weigh the sample (up to 19 g) into an Infrasil cell (4-cm path length), flush the cell with dry air and fill it with Br (dried by means of molecular sieve 4A) and shake for 1 h. Measure the absorbance of this soln. and of a Br blank at 3665 cm^{-1}. The coeff. of variation (10 results) in the determination of 54 μg g^{-1} of H_2O in a ground sample was 4·3%. R. W.

1B189. **Fluorescence determination of rhenium in ores and alloys.** Pilipenko, A. T.; Volkova, A. I.; and Shevchenko, T. L. (Inst. Colloid and Water Chem., Acad. Sci. Ukr. SSR, Kiev). *Ukr. Khim. Zh.* (*Russ. Ed.*), 1977, **43** (6), 653–654 (in Russian).—Safranine T (C.I. Basic Red 2) forms a bright-orange fluorescent compound with ReO_4^- at pH 4 to 8. The sample (0·1 g) is fused with 1 g of Na_2O_2 - NaOH (1 : 1), and the cooled melt is dissolved in aq. 20% NaOH (50 ml). This soln. is shaken with acetone (10 ml), and, after separation of the phases, the organic layer is washed with the NaOH s'n. (10 ml) (to remove traces of Mo, W, and V). The acetone soln. is then evaporated to dryness, the residue is dissolved in H_2O (20 ml), and this soln. is mixed with aq. mM dye (0·5 to 1 ml). The pH of the mixture is adjusted to 6 with dil. H_2SO_4, and the Re complex is extracted into dichloroethane (6 ml) for fluorimetry at 546 nm. The method is sensitive to 0·02 μg of Re in 5 ml of extract. A. B.-S.

1B190. **Potentiometric titration of rhenic acid in various concentrations.** Sinyakova, G. S. *Latv. PSR Zinat. Akad. Vestis, Kim. Ser.*, 1977, (1), 61–63 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (16), Abstr. No. 16G20.

1B191. **Simultaneous coulometric determination of rhenium and molybdenum in alloys.** Kostromin, A. I.; Evgen'ev, M. I.; and Novikova, L. A. (V. I. Ul'yanov-Lenin Kazan State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1162–1165 (in Russian).—Microgram amounts of Re^{VII} can be titrated with electro-generated Cr^{II} (*cf. Anal. Abstr.*, 1974, **27**, 3847) in M-HCl as the supporting electrolyte with use of two platinum electrodes (at a p.d. of 0·6 V) for end-point indication. Up to 200-fold amounts (relative to Re) of Co and Ni, 150-fold amounts of Cr^{III}, 10-fold amounts of W^{VI}, V^V, Nb^V and Mo^{VI} and 25-fold amounts of Fe^{III} do not interfere if the supporting electrolyte contains at least 10-fold molar amounts of NaF and Na_3PO_4 relative to the foreign metals. Any Mo^{VI} present is titrated after the Re^{VII}; in this event the titration curve exhibits two distinct end-points. The method has been used for determining 47% of Re and 53% of Mo in a Re - Mo alloy, and 2% of Re and 6·4% of Mo in an alloy also containing Si, Mn, Cr, Ni, Co and Fe. The coeff. of variation was <6% (6 results) for Re and <5% for Mo. M. P.

1B192. **Use of atomic-absorption and atomic-emission spectrometry in analysing high-purity substances. Analysis of rhenium.** Zelentsova, L. V.; Yudelevich, I. G.; and Shaburova, V. P. (Inst. Inorg. Chem., Acad. Sci. USSR, Siberian Branch, Novosibirsk). *Zh. Anal. Khim.*, 1977, **32** (6), 1166–1170 (in Russian).—Traces of Ag, Al, Bi, Ca, Cd, Cr, Cu, Fe, Mg, Mn, Ni, Pb and Zn were determined by a.a.s. with use of a graphite-furnace atomiser; traces of K, Li and Na were determined by a.e.s. with an acetylene - air flame. The sample (50 mg) was dissolved in conc. HNO_3 (1 ml), and H_2O (4 ml) was added. For a.a.s., a 20 to 50-μl portion was injected into the graphite furnace, dried at 100° for 60 s, ashed at 600° to 1450° (according to the element to be determined) for 35 to 85 s, and atomised at 1900° (for Zn, Cd and Bi) to 2700° (for Cr and Al). Limits of determination ranged from 5 p.p.b. (for Cd) to 1 p.p.m. (for Bi), but could be improved (to 0·4 p.p.b. for Cd or 2 p.p.b. for Bi) by using a 1-g sample and preliminary extraction of the Re with 0·3M-trioctylamine *N*-oxide in toluene; in this instance the sample was dissolved in aq. 30% H_2O_2. The coeff. of variation was <30% (2 to 7 results) for either method. M. P.

Group VIII

Fe, Co, Ni, Ru, Rh, Pd, Os, Ir, Pt

1B193. **Analytical applications of heteropolymolybdates: the iron(II) - ammonium molybdate reaction.** Sanchez Perez, A.; Polo Diez, L.; and Gonzalez Perez, C. (Dept. Quim. Anal., Fac. Cienc., Univ. Salamanca, Spain). *Quim. Anal.*, 1977, **31** (1), 19–22 (in Spanish).—In some slightly acid media, Fe^{II}

reacts with $(NH_4)_6Mo_7O_{24}$ to give unstable red, then blue, colours, but in H_2SO_4 a blue colour with an absorption max. at 590 nm is formed; this is suitable for the spectrophotometric determination of Fe^{II}. Optimum conditions are a medium 0·2M in the molybdate and M in H_2SO_4; Beer's law is obeyed for Fe^{II} concn. from 40 to 250μM. L. A. O'N.

1B194. **Analytical application of oleophilic quaternary ammonium salts. XI. Extraction of the iron(III) - catechol violet - tridodecylethylammonium ternary complex.** Shijo, Yoshio (Dept. Ind. Chem., Fac. Eng., Univ., Ishii-machi, Utsunomiya, Japan). *Bull. Chem. Soc. Jpn.*, 1977, **50** (4), 1013–1014.—Tervalent Fe (0·2 to 3μM) is extracted at pH 5·8, from an aq. medium 0·04mM in catechol violet, into a mM soln. of tridodecylethylammonium bromide in isobutyl methyl ketone by shaking for 5 min. The absorbance of the organic layer is measured at 623 nm. A rectilinear calibration graph is obtained over the range stated. Interference from Cu, Bi and Sn is masked by 2-mercaptoethanol and that from Th, U and Al by H_2O_2 - KF. However, V, Mo, W, Cr, Zr, NO_3^-, I^-, SCN^- and ClO_4^- interfere. The composition of the complex is discussed. (For Part X see *Anal. Abstr.*, 1978, **34**, 1B163.) E. A.

1B195. **Iron(III) - iron(II) ratios in bulk rock samples by Mossbauer spectroscopy. Determination on standard rock samples G-2, GA, W-1 and MICA-Fe.** Bancroft, G. M.; Sham, T. K.; Riddle, C.; Smith, T. E.; and Turek, A. (Dept. Chem., Univ. Western Ontario, London, Canada). *Chem. Geol.*, 1977, **19** (4), 277–284.—Room-temperature ^{57}Fe Mossbauer spectra have been recorded for the standard rocks cited, and results for the ratio of Fe^{III} to Fe^{II} by chemical methods and Mossbauer spectroscopy are compared. The errors involved in each method are assessed. For rocks low in Fe, Fe^{III} appeared to be considerably over-estimated by the chemical method, although the spread of individual laboratory values tended towards a low limit close to the value obtained by the Mossbauer technique.
INIS Atomindex

1B196. **Coulometric determination of iron in hydrochloric acid pickling baths.** Gantner, Hans (Anal. Lab., von Moos Stahl A.-G., Lucerne, Switzerland). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 39–40 (in German).—A 5-ml sample is heated with H_2SO_4 to expel HCl. The mixture is diluted to 500 ml, and the Fe^{III} in a 5-ml portion is reduced to Fe^{II} by a rotating (1000 r.p.m.) cadmium rod (7·5 cm × 1 to 1·5 cm) in a vessel of i.d. $\simeq$5 mm larger than the diam. of the rod; treatment for $\simeq$2 min suffices. Then 15 ml of 4% $Ce_2(SO_4)_3.5H_2O$ soln. in 15% H_2SO_4 is added, and the titration is carried out at constant current in a vol. of 25 ml with a Metrohm E 524 coulostat with use of a platinum-black generating electrode separated from the cathode [in a separate compartment containing 15% $(NH_4)_2SO_4$ soln.] by a glass frit; the end-point is detected with a platinum - silver/AgCl electrode pair connected to an E 516 Titriskop. H. L. L.

1B197. **Diffractionless X-ray spectrographic determination of small amounts of calcium [in iron ore].** Kochmola, N. M.; Kalosha, V. K.; and Yuksa, L. K. (Kommunarsk Mining and Metall. Inst., USSR). *Zavod. Lab.*, 1977, **43** (6), 691–692 (in Russian).—An apparatus is described for determining up to 1·5% of CaO in coarse granular iron ore. The CaO content is determined by measuring (with an Ar - CH_4 proportional counter) the secondary X-rays excited by radiation from a chromium-target tube; operating conditions are indicated. The time required for sample preparation (which can be automated) and analysis is $\simeq$3 min. A. S. W.

1B198. **Determination of non-metallic inclusions in metallic materials after ultrasonic fracture.** Mitsche, R.; and Stanzl, S. (Montanuniv. Leoben, Austria). *Mikrochim. Acta*, 1977, Suppl. VII, 531–537 (in German).—Inclusions in cast iron and steel can be revealed by brittle fracture produced ultrasonically and identified directly by scanning electron microscopy. J. W. P.

1B199. **Influence of the matrix in energy-dispersive X-ray analysis of inclusions and precipitates [in steels].** Pohl, M.; and Burchard, W.-G. (Gemeinschaftslabor Elektronenmikroskopie RWTH, Aachen, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 261–278 (in German).—Suitable acid mixtures (*e.g.*, H_3PO_4 - H_2O_2, HCl - HNO_3 - ethanol, and $HClO_4$ - acetic acid) are cited for chemical and electrolytic deep-etching of steel samples to reveal particles of oxides, sulphides or carbides. The relationship between particle size of the matrix and degree of interference is discussed, and a two-stage preparation procedure to overcome this interference is described. J. W. P.

1B200. **Surface investigations of metals and alloys by ESCA [X-ray photo-electron spectroscopy].** Holm, R.; and Storp, S. (Bayer A.-G., Leverkusen, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 139–152 (in German).—The method has been used to study the composition of oxide films on stainless steels and the effect of Zn on the formation of oxide layers on a Ag - Sn alloy. The effect of ion bombardment on the surfaces of various steels is discussed. J. W. P.

1B201. **Determination of carbon and nitrogen concentration gradients in steel by nuclear methods.** Engelmann, C.; Loeuillet, M.; and Coliniaux, M. (Dept. Rech. Anal., CEN Saclay, Gif-sur-Yvette, France). *Mem. Sci. Rev. Metall.*, 1976, **73** (10), 611–623 (in French).—Nuclear techniques for determining C and N in steel are briefly reviewed. The procedures recommended involve proton and deuteron activation to induce the reactions ^{12}C-(p,γ)^{13}N, ^{15}N(p,$\alpha\gamma$)^{12}C, ^{12}C(d,p)^{13}C and ^{14}N(d,α)^{12}C. The relative error when determining C or N by deuteron activation was 2%. Chem. Abstr.

1B202. **Determination of silicon in high-alloy steel by 14-MeV neutron activation and high-resolution gamma-ray spectrometry.** Chiba, Minoru (Natl. Res. Inst. Met., Tokyo, Japan). *Trans. Natl. Res. Inst. Met. (Tokyo)*, 1976, **18** (4), 133–138.—A rapid non-destructive method for determining Si in such samples was studied, with use of Ba as internal standard; the radioactive nuclides produced were measured with a Ge(Li) detector. To determine Si, the 1·78-MeV γ-ray of ^{28}Al [the product of the ^{28}Si(n,p) reaction] was measured, and, for the internal standard, that of the 0·662-MeV ray of ^{137m}Ba produced by the ^{138}Ba(n,2n) reaction. Interference by the 1·81-MeV γ-ray of ^{56}Mn [from Fe and/or Co in the sample by the ^{56}Fe(n,p) and ^{59}Co-(n,α) reactions] was prevented by using high-resolution γ-ray spectrometry and an advanced total-peak-area method for processing the results. The adaptability of ^{137m}Ba as internal standard for ^{28}Al was investigated by experiments involving use of various neutron fluxes; the calculated results were

accurate to within <1%. Results by the proposed method differed by <±5% from those of conventional methods and the reproducibility was of the same order as the statistical error of the radioactivity measurements. INIS ATOMINDEX

1B203. **Determination of nitrogen in high-speed cutting steel with use of pulsed heating.** Strelkov, V. V.; Kuznetsov, L. B.; Zaitsev, A. V.; and Rodionova, E. P. (Steel and Alloy Inst., Moscow, USSR). *Zavod. Lab.*, 1977, **43** (6), 660–662 (in Russian).—Complete release of N from such steel is difficult to attain, owing to formation of nitrides. The following conditions are recommended for steels containing ≃0·033% of N—temp., 2500°; pulse time, 10 s; the addition of Ni (20% of the sample wt.) slightly enhances the degree of release of N. A. S. W.

1B204. **Determination by atomic-absorption spectrophotometry of arsenic, selenium and tin in metallurgical specimens.** Condylis, A.; and Hocquaux, H. (Creusot-Loire, Centre Rech. Unieux, Firminy, France). *Analusis*, 1977, **5** (5), 228–235 (in French).—The optimum wavelengths and the relative performance of hollow-cathode and electrodeless-discharge lamps for the determination of Sn, As and Se in steel were investigated. Corrections for the effect of non-specific absorption and for the presence of various ions and organic solvents were determined. During dissolution of the sample, addition of HF prevents subsequent interference from refractory elements (*e.g.*, Ti, Nb, W and Si) in the determination of As and the presence of an excess of HNO_3 prevents the formation of volatile hydrides and chlorides. B. D. H.

1B205. **Determination of bismuth in steels and cast irons by atomic-absorption spectrophotometry with an induction furnace: direct analysis of solid samples.** Andrews, David G.; and Headridge, James B. (Dept. Chem., Univ., Sheffield, England). *Analyst* (*London*), 1977, **102** (1215), 436–445.—Details are given of an improved version of an induction furnace previously described by Headridge (*Anal. Abstr.*, 1974, **27**, 620) and of its use, in conjunction with a Perkin-Elmer 300 S atomic-absorption spectrophotometer, for the determination of Bi. A sample of steel or iron (1 to 12 mg) is introduced via the double-valve system into the heated graphite furnace, and after the atomic-absorption peak has been recorded, Ar is passed to purge the furnace; samples can be introduced at intervals of 2 to 5 min. Down to 0·004 p.p.m. of Bi could be detected, and at the 0·04-p.p.m. level the coeff. of variation was <10%. R. W.

1B206. **Determination of oxygen in complex steels and materials with high vapour pressure.** Kuznetsov, L. B. (Steel and Alloy Inst., Moscow, USSR). *Zavod. Lab.*, 1977, **43** (6), 663–664 (in Russian).—The use of a special graphite crucible with several tightly abutting graphite sleeves fixed to the top, similar to a cascade crucible, is recommended. The sample is placed in the bottom of the crucible, with or without a nickel - tin bath depending on the sample, and heated to 1600°. A high degree of release of O is achieved, and errors due to sorption of oxides of C in sublimed metal are avoided. The method is satisfactory for Ni - Cr and Cr - Mn steel containing ≃0·018% of O. A. S. W.

1B207. **Gas analysis with a small mass spectrometer. II. Determination of oxygen and nitrogen in steel.** Koch, O. G. (Chem. Lab., Neunkircher Eisenwerk A.-G., Neunkirchen/Saar, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 13–18 (in German).—An apparatus is described for melting the sample and extracting the gases, which are passed to an AEI MS 10 mass spectrometer. Samples (≃0·7 g) of steel are heated in a 45-A d.c. arc for 25 s in an atmosphere of Ar. Down to 10 p.p.m. of O and 3 p.p.m. of N can be detected, and coeff. of variation at intermediate levels are ±3 to 5% for O and ±3 to 8% for N. (For Part I see *Anal. Abstr.*, 1969, **16**, 38.) H. L. L.

1B208. **Chemical analysis for metallic chromium and iron in semi-reduced chromium pellets [from a ferrochromium plant].** Okuda, Kinnosuke; Muranaka, Mitsugu; Takeda, Kikuo; Abe, Hiroshi; Sakurai, Ryoji; and Goto, Kazuyoshi (Japan Metals and Chemicals Co. Ltd., Tokyo, Japan). *Tetsu To Hagane*, 1977, **63** (2), 338–344 (in Japanese).—Metallic Cr and Fe in such pellets form a complex carbide, which is easily dissolved in conc. H_2SO_4; however, this acid also dissolves some Cr and Fe from unreacted chromite ore in the pellet, leading to high results for metallic Cr and Fe. Use of H_2SO_4 (1 : 3) gives full recovery of metallic Cr and Fe without dissolution of the chromite. The sample should be heated under reflux for 30 min with the dil. acid, which boils at ≃111°. The proposed method gave results without bias in four laboratories; for metallic Cr, the standard deviation was ±0·15% for a mean value of 13·6%, and for metallic Fe it was ±0·20% for a mean value of 11·0%. CHEM. ABSTR.

1B209. **New catalytic reaction for determination of cobalt(II) ion traces.** Ionescu, G.; Duca, A.; and Aelenei, N. (Dept. Phys. Chem., Polytech. Inst., Iasi, Romania). *Bul. Inst. Politeh. Iasi, Sect.* **2**, 1976, **22** (1–2), 23–29.—The kinetic determination was effected by measuring the rate of autoxidation (catalysed by Co^{2+}) of 1·6mM-gallic acid at pH 10·33. The calibration graph was rectilinear for 0·08 to 0·8μM concn. of Co^{2+}, and the relative errors were <4·3%; Ag^+, Ni^{2+}, Fe^{3+} and CN^- interfered. CHEM. ABSTR.

1B210. **Spectrophotometric determination of cobalt(II), nickel(II) and copper(II) with 1-(2-quinolylazo)-2-acenaphthylenol.** Singh, Ishwar; Garg, B. S.; and Singh, R. P. (Dept. Chem., Univ. Delhi, India). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 42.—To the sample soln., containing 2 to 9 μg of Co, 6 to 23 μg of Ni or 2·4 to 14 μg of Cu, are added an excess of a 2mM soln. of the reagent in methanol and 2 to 4 ml of 2M-Na acetate. From this soln. [made 50% (v/v) in methanol and of vol. 10 ml for Co or Cu or 25 ml for Ni] the complex is extracted into 10 ml of CCl_4, and the absorbance is measured at 550 nm for Co, 560 nm for Ni or 545 nm for Cu. Cyanide, PO_4^{3-}, citrate, edta, Fe^{II} and Zn interfere; a list of other interfering species is presented. H. L. L.

1B211. **Micro-chromatographic semi-quantitative determination of nickel in alloys.** Patroescu, Constantin; and Duican, Maria. *Rev. Chim.* (*Bucharest*), 1977, **28** (5), 485–486 (in Romanian).—Dissolve 0·1 to 0·5 g of alloy in 12 ml of HCl - HNO_3 (3 : 1), evaporate almost to dryness, add 2 ml of conc. H_2SO_4, and heat to fumes; cool, dilute, neutralise with aq. NH_3, and dilute to 100 ml (pH 6 to 7). Dilute an aliquot of this soln. to 5 ml, and introduce into the soln. a glass tube (25 cm × 0·2 cm) closed with a filter-paper plug at one end and containing a 10-cm length of silica gel supporting dimethylglyoxime (**I**) (mix 20 g of silica gel with

10 ml of ethanolic 1% **I**, dry in an oven for 2 h, and grind). Ascertain the Ni content of the soln. from the height of the pink layer, with reference to a calibration graph. For a sample known to contain 78·89% of Ni (determined gravimetrically with **I**), results of 81·05, 77·57, 75·62 and 76·55% were obtained by the proposed method. C. D. K.

1B212. **Extraction of nickel(II) in aqueous thiocyanate - perchlorate solutions with trioctylphosphine oxide in hexane and with 4-methylpentan-2-one.** Murai, Ryokichi; Iwahori, Shigehisa; and Sekine, Tatsuya (Dept. Chem., Sci. Univ., Kagurazaka, Shinjuku-ku, Tokyo, Japan). *Bull. Chem. Soc. Jpn.*, 1977, **50** (5), 1315–1318.—Nickel (0·01 to 10mM) or Co (0·1 to 10mM) was extracted from aq. soln. of $NaSCN - NaClO_4$ (up to M in either salt) into an equal vol. of hexane, containing 0·005 to 0·08M-trioctylphosphine oxide or 4-methylpentan-2-one, by rotating the soln. for 2 or 24 h (for Co or Ni, respectively). The metal in the organic phase was re-extracted into 0·1M-HCl and determined by a.a.s. The results were used in predicting the structures of the complexes. R. P. E.

1B213. **Amperometric titration of mixtures of nickel with other elements.** Zhdanov, A. K.; and Barkhudar'yan, A. A. (Tashkent State Univ., USSR). *Dokl. Akad. Nauk Uzb. SSR*, 1976, (2), 56–58 (in Russian).—To analyse mixtures of Ni with Fe, Cu or Ag and with Cu plus Ag, the Fe and Ni were titrated with edta at pH 1 to 2 and 4·5 to 5·0 and at 1·2 and 1·0 V, respectively. The sum of Ni and Cu was titrated with edta at pH 4·5 to 5·0; in another aliquot, Ni was titrated selectively with dimethylglyoxime at 0·8 V in ammoniacal buffer medium of pH 9 to 10, and the Cu was found by difference. Silver was titrated with KI in acid medium at 0·8 V; Ni in the presence of Ag was titrated with edta. For the ternary mixture, in one aliquot Ag and the sum of Ni and Cu were determined, and in another Ni was determined by titration with dimethylglyoxime. The errors were $<2·60\%$ when determining 0·06 to 12 mg of a metal.

CHEM. ABSTR.

1B214. **Spectrographic determination of iron and cobalt in powdered specimens based on nickel(III) oxide.** Catasus-Portuondo, M.; and Fernandez-Bulanos, J. *Vestn. Leningr. Univ.*, 1977, (4), *Fiz., Khim.*, (1), 62–68 (in Russian).—Nickel oxide concentrates were analysed for impurities of Co and Fe by emission spectrography (10-A a.c. arc between graphite electrodes); the samples were diluted 1 : 2 with graphite, and the intensities of the Co and Fe lines were measured at 307·234 and 304·760 nm, respectively, *vs.* the Ni line at 310·771 nm. Results were less than those of chemical methods by 18 and 9% for 0·62 to 1·09% of Co and 0·16 to 0·41% of Fe, respectively; it is considered that the chemical methods gave high values in the presence of Ni in excess. CHEM. ABSTR.

1B215. **Micro-analytical investigations of high-temperature alloys for gas-turbine blades.** Thien, Volker; Schieferstein, Udo; and Voss, Wolfgang (Kraftwerk Union A.-G., Abt. Tech. Werkstoffe/Metall., Muelheim/Ruhr, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 389–403 (in German).—A combination of an energy-dispersive system (EDAX V) with a scanning electron microscope was used to identify a Zr - S phase in a Ni - Cr - Co alloy and to examine the effect of heat treatment on the composition and structure of a Cr - Al coating on another Ni - Cr - Co alloy. J. W. P.

1B216. **Indirect polarographic determination of thiourea in nickel-plating electrolyte by means of a catalytic pre-wave in the nickel(II) - thiourea system.** Ruvinskii, O. E.; and Kulaeva, L. T. (Krasnodar Polytech. Inst., USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1256–1259 (in Russian).—The method is based on the catalytic effect of thiourea on the reduction of Ni^{II} at a dropping-mercury electrode. Under optimum conditions (*viz*, with a supporting electrolyte of pH 6 that is 0·4M in $NiSO_4$, 4M in $NaNO_3$, M in Na acetate and 3mM in tetraethylammonium iodide), the catalytic current measured at $-0·75$ V *vs.* the n.c.e. is rectilinearly related to concn. of thiourea in the range 10μM to mM. The coeff. of variation was 4% (7 results) when determining 0·07mM-thiourea in a plating electrolyte containing $NiSO_4$, $NiCl_2$ and H_3BO_3. M. P.

1B217. **Spectrophotometric determination of ruthenium in nitroso-complexes by using photolysis.** Nikol'skii, A. B.; and Popov, A. M. (Leningrad State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 999–1003 (in Russian).—The method involves photo-decomposition of such complexes as $K_2RuNOCl_5$ (**I**) or $K_2RuNOBr_5$ (**II**) in HCl media to form aquo-acido-complexes of Ru^{III}, or in neutral or basic media to form hydroxo-acido-complexes of Ru^{IV}; the aquo- and hydroxo-complexes can be determined spectrophotometrically by using conventional reagents. To determine 0·1 to 0·35mM-**I** or **II**, the test soln. is made 6M in HCl and irradiated for 15 min with a mercury lamp, then 2 ml of this soln. is heated with aq. 10% thiourea (0·5 ml) at 50° for 10 min. The absorbance of the Ru^{III} - thiourea complex is measured at 650 nm ($\epsilon = 2650$). The coeff. of variation was $<0·7\%$ (5 results) when determining $\simeq 0·1$ to 0·2mM-**I** or **II**. If photolysis of **I** or **II** is carried out in dimethyl sulphoxide medium, coloured complexes of Ru^{II} ($\epsilon \simeq 4000$) are formed; these complexes can be used for direct spectrophotometric determination of Ru. M. P.

1B218. **Organic reagents for noble metals. XII. Reactions between rhodium(I) and reagents of the allthiox [8-(allylthio)quinoline] type.** Dedkov, Yu. M.; Ermakov, A. N.; Kotov, A. V.; and Slotintseva, M. G. (V. I. Vernadskii Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (5), 870–875 (in Russian).—The stability and structures of the 1 : 1 and 2 : 1 complexes formed by allthiox with Rh^I have been studied by u.v., visible and i.r. spectrophotometry. The absorption spectra of these complexes exhibit a shoulder between 350 and 450 nm, the ϵ value for the 2 : 1 complex being $\simeq 10{,}000$ at 400 nm. Both the sensitivity and the colour contrast of the reaction can be improved by introducing an electron-attracting group (*e.g.*, a sulpho-group) into position 5 of the quinoline ring. M. P.

1B219. **Analytical applications of some nitroanisaldoximes : 2-nitro-*p*-, 4-nitro-*o*- and 5-nitro-*o*-anisaldoximes. II. Quantitative applications.** Buscarons, F.; and Plana, R. (Dept. Quim. Anal., Fac. Cienc. Quim., Univ. Barcelona, Spain). *Quim. Anal.*, 1977, **31** (1), 27–29 (in Spanish).—To determine Pd^{II}, the test soln. in acetic acid is treated, at 50° to 60°, with a 0·5% soln. of any of these aldoximes in ethanediol (added in excess). The ppt. is filtered off, washed with hot water, dried and weighed. Bivalent Cu can be determined similarly, except that aq. NH_3 is added to the test soln. to give pH 9 before addition of the reagent. L. A. O'N.

1B220. **Spectrophotometric titration of palladium with edta using some Schaeffer β-acid azo-dyes as indicators.** Khalifa, H.; Issa, Y. M.; and Abdel-Ghani, N. T. (Chem. Dept., Fac. Sci., Cairo Univ., Giza, Egypt). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 46.—To equal vol. of soln. each containing 31 to 106 μg of Pd add 1 to 1·5 ml of 0·1% 5-(4-chlorophenylazo)-6-hydroxynaphthalene-2-sulphonic acid soln., incremental vol. of mM-edta and 5 ml of universal buffer soln. of pH 5 or acetate buffer soln. of pH 6, dilute to 10 ml so that each final soln. contains 15% of isopropyl alcohol, and measure the absorbances at 560 nm. The 5-(4-sulphamoylphenylazo)-, 5-(4-bromophenylazo)- and 5-(4-iodophenylazo)-analogues were also found to be useful for the range 21 to 64 μg. H. L. L.

1B221. **Reaction of different forms of palladium with nitrosulphophenol M [3-(2-hydroxy-5-nitro-3-sulphophenylazo)-6-(3-sulphophenylazo)chromotropic acid].** Gur'eva, R. F.; Savvin, S. B.; Romanov, P. N.; Akimova, T. G.; and Syanava, E. M. (V. I. Vernadskii Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (5), 1009–1015 (in Russian).—It is shown that all sol. forms of Pd^{II} or Pd^{IV} [nitrato-, hydroxo-, aquo- and chloro-complexes (except $PdCl_3^{3-}$ and $PdCl_6^{4-}$), hydrolysed polymeric species, and all types of mixed-ligand complexes] react identically with the reagent cited (**I**) to form a blue Pd^{II}-**I** complex (Pd^{IV} is reduced to Pd^{II} during complex formation); the reaction can be carried out at any acidity from that of M-HNO_3, M-H_2SO_4 or M-H_3PO_4 to pH 6. The complex has max. absorption at 625 nm (ϵ = 80,000), and Beer's law is obeyed for 0·08 to 2·4 μg ml^{-1} of Pd; <500-fold amounts (relative to Pd) of Ag or La and 100-fold amounts of Cu, Bi, Ni or Al do not interfere; Fe^{III} can be masked with H_3PO_4. A method based on these findings has been used for determining 0·005 to 1% of Pd in alloys with Ag. The samples (0·1 to 1 g) were dissolved in HNO_3, and the colour reaction was carried out in a medium 0·5M in HNO_3, 0·4N in H_3PO_4 and containing 40 p.p.m. of **I**. The coeff. of variation was <9% (12 results). M. P.

1B222. **Adsorption-polarised electrodes for end-point indication in potentiometric titrations. IV. Determination of palladium as iodide.** Bartels, U.; and Umland, F. (Anorg. Chem. Inst., Wilhelms Univ., Muenster, Westf., W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **284** (4), 263–266 (in German).—Palladium (2·5 to 25 p.p.m.) is determined by potentiometric titration with mM- or 10mM-KI, with end-point indication by means of adsorption-polarised electrodes. The electrodes are prepared by coating platinum-foil electrodes with palladium or platinum by electrolysis for 30 s at 1 A, and a platinum-foil counter-electrode is used. The end-point appears as a sharp positive potential peak, and optimum indication is obtained in HCl (pH 2) or 0·5M-H_2SO_4 medium. The coeff. of variation for 50 μg of Pd is 1·07% (15 determinations). The method has been applied to analysis for Pd in catalysts based on calcium carbonate or carbon. Large amounts of Ir^{IV} interfere, and Au^{I} and Ag are co-titrated in H_2SO_4 medium; Au is therefore reduced to the metal with SO_2, and Ag is removed by pptn. as AgCl. (For Part III see *Anal. Abstr.*, 1977, **32**, 4A12.) C. K. L.

1B223. **Use of electrophoresis in analytical chemistry. II. Effect of the carrier buffer electrolyte on electrophoretic behaviour of ions.** Luginin, V. A.; and Tserkovnitskaya, I. A. *Probl. Sovrem. Anal. Khim.*, 1976, **1**, 162–168 (in Russian).—The effect of impregnating the carrier paper with various reagents on the electrophoretic separation of Pd^{II} and Pt^{IV} was studied. These species were separated by electrophoresis in 0·1M-HCl or -HBr on papers impregnated with 2% dimethylglyoxime soln. in ethanol or with up to 10% tributyl phosphate soln. in benzene, respectively. With tributyl phosphate, Pd migrated to a slight extent, but with dimethylglyoxime it remained at the origin; Pt migrated rapidly to the anode, and ions such as Fe^{III}, Co^{II} or Cu^{II} migrated to the cathode. Impregnation of the paper with trioctylamine soln. in benzene resulted in tailing of the analyte zones. (For Part I see *Anal. Abstr.*, 1978, **34**, 1B9.) CHEM. ABSTR.

1B224. **Solvent extraction of complexes of platinoids and copper with diphenylthiourea.** Petrukhin, O. M.; Shevchenko, V. N.; Zakharova, I. A.; and Prokhorov, V. A. (V. I. Vernadskii Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (5), 897–903 (in Russian).—Bivalent Pd, Pt^{II}, Ru^{II}, Rh^{II}, Os^{II} and Cu^{I} were extracted from aq. medium 6M in HCl and 10mM in $SnCl_2$ into 8·8mM-diphenylthiourea (**I**) in $CHCl_3$; Co, Ni, Mn, Fe and Zn were not extracted under these conditions. The compositions of the extracted species are discussed. The complexes could be partly separated by t.l.c. on alumina, with $CHCl_3$ as mobile phase; the Pd^{II}, Pt^{II}, Rh^{II} and Ru^{II} species each gave a single spot, but the Os^{II} complex gave three. M. P.

1B225. **Thin-layer chromatography of noble elements. XI. Behaviour and separation of phosphate complexes of platinoids in aqueous phosphoric acid medium.** Volynets, M. P.; Ermakov, A. N.; Ginzburg, S. I.; Chalisova, N. N.; Gur'eva, R. F.; Dubrova, T. V.; Yuz'ko, M. I.; and Fomina, T. A. (V. I. Vernadskii Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (5), 914–921 (in Russian).—The chemistry of such complexes of Pt, Ir, Pd and Rh in H_3PO_4 medium has been studied spectrophotometrically and by t.l.c. on silica gel, cellulose and Dowex 50-X8 resin. The tendency of the complexes to be hydrolysed in M to 3M-H_3PO_4 decreased in the order Pd, Ir, Rh, Pt. Binary mixtures of Pd with Pt, Rh or Ir could be separated into their constituents by t.l.c. *Procedure*—The test soln. (2 μl), 10mM in the platinoids and M to 16M in H_3PO_4, was applied to a plate coated with Dowex 50-X8 resin (Na^+ form), and the plate was set aside for 5 h for hydrolysis of the Pd^{II} species. The chromatogram was then developed with 0·01M-H_3PO_4, dried at room temp., sprayed with 0·2% chlorsulphophenol-azo-rhodanine [5-chloro-2-hydroxy-3-(4-oxo-2-thioxothiazolidin-5-ylazo)benzenesulphonic acid] soln. in 15M-acetic acid and heated at 70° for 1 h; the platinoids were then visible as pink spots. The lower limit of detection was ≃50 ng of platinoid per spot. (For Part X see *Anal. Abstr.*, 1977, **33**, 5B178.) M. P.

1B226. **Thioantipyrine and some of its derivatives as analytical reagents for osmium.** Akimov, V. K.; Busev, A. I.; and Kliot, L. Ya. (Sci.-Res. Inst. Org. Intermediates and Dyes, Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 1004–1008 (in Russian).—Thioantipyrine (**I**), bis(thioantipyrinyl)methane and 1,1-bis(thioantipyrinyl)butane have been studied as spectrophotometric reagents for Os^{VIII}; **I** is considered the most suitable, as it forms a 1 : 4 (metal to **I**) complex in a medium 0·2 to 4M in HCl. This

complex exhibits max. absorption at 735 nm (ϵ = 8600), and Beer's law is obeyed for 1 to 20 μg ml^{-1} of Os (a >30-fold molar excess of **I** relative to Os is needed). There is no interference from the amounts (relative to Os) shown in parentheses of the following species—Co, Mn^{II}, Al or Mg (1000), Zn, Ga, Ti^{IV} or Ni (300), Cd or Cr^{III} (50), Ru^{IV} (30), Rh^{III} (12), Fe^{III}, Hg^{II} or Pd^{II} (6) or Pt^{IV} (1). The coeff. of variation was 1·2% (7 results) when determining 12 μg ml^{-1} of Os in a prepared soln. An Os^{VIII} - **I** - trichloroacetate (1:2:2) complex is extractable from a medium 0·2 to 4M in HCl and >50mM in trichloroacetic acid into $CHCl_3$; this complex exhibits max. absorption at 590 nm (ϵ = 6200), and Beer's law is obeyed for 1·5 to 30 μg ml^{-1} of Os; a 400-fold amount of Ru^{IV} does not interfere.

M. P.

1B227. **Determination of platinum in sands by flameless atomic absorption using the HGA-2100.** Tello, A.; and Sepulveda, N. [Centro de Investigacion Minera y Metal. (CIMM), Santiago, Chile]. *At. Absorpt. Newsl.*, 1977, **16** (3), 67–69.—Platinum was determined in the range 0·1 to 1·5 μg g^{-1} in sand by flameless a.a.s. at the Pt 265·9-nm line (0·7 nm slit width) after cupellation to concentrate the Pt and separate it from interfering elements. The sample (10 g, <100 mesh) was first fused for 2 h at 1200° with 90 g of flux [PbO - Na_2CO_3 - $Na_2B_4O_7$ - flour (3·5:4:1:0·3)]. The melt was cooled in an iron vessel, and the regulus was separated from the slag. The regulus was heated at 925° to volatilise the Pb, the residue was warmed with 1 ml of aqua regia and, after dissolution, the vol. was made up to 25 ml with H_2O. Then 20-μl portions of the soln., containing Pt in the range 0·02 to 1·0 μg ml^{-1}, were injected into the graphite furnace, dried at 105° for 35 s, charred at 800° for 10 s, and atomised at 2700° for 8 s. The sensitivity was 590 pg for an absorbance of 0·004, the average deviation of the peak height was 6·5% at the 0·1-μg-ml^{-1} level and 5% at the 1-μg-ml^{-1} level, and recovery tests showed that the method gave acceptable accuracy. C. K. L.

1B228. **Spectrophotometric determination of rhodium and palladium in platinum alloys.** Duchkova, Hana; Cermakova, Ludmila; and Malat, Miroslav (Dept. Anal. Chem., Charles Univ., Prague, Czechoslovakia). *Chem. Listy*, 1977, **71** (5), 535–537 (in Czech).—The sample was decomposed with aqua regia, the soln. was evaporated, and the residue was dissolved in HCl. The Pd was adsorbed on Amberlite IRC-50 resin (Na^+ form) from 0·1M-HCl medium. The percolate, containing Pt and Rh, was treated with 1 g of NaCl and applied to Dowex 2 resin (Cl^- form), from which Rh was eluted with 50 ml of 2M-HCl and Pt with 100 ml of 7M-HCl. A portion (0·5 ml) of either eluate was diluted to 10 ml, and to a 1-ml aliquot were added 2·5 ml of mM-Eriochrome cyanine R (C. I. Mordant Blue 3), 0·5 ml of 10mM-cetylpyridinium bromide and 2·5 ml of 0·2M-acetate buffer of pH 3·4. The mixture was heated for 50 min on a boiling-water bath, then diluted to 25 ml, and the absorbance was measured at 630 nm against a reagent blank. The Pd was eluted from the Amberlite with 50 ml of M-HCl and determined similarly, except that a buffer soln. of pH 4·5 was used and the reaction mixture was heated for 20 min at 50°. J. Z.

1B229. **Determination of gold in platinum - rhodium - gold alloys using a neutron generator.** Pelekis, L.; and Jurka, J. (Inst. Fiz., Riga, Latv. SSR). *Latv. PSR Zinat. Akad. Vestis, Fiz. Teh. Zinat. Ser.*, 1976, (4), 6–11 (in Russian).—From ≃0·1 to 5% of Au was determined in such alloys (used for the manufacture of heat-resistant jets in glass-fibre production) by non-destructive n.a.a. with use of 14-MeV fast neutrons; possible errors in the analysis are discussed, and simple expressions are derived for the calculation of correction factors for measurement geometry, γ-ray attenuation, and neutron shielding by the sample material where there are differences in thickness and area of samples and standards. *Procedure*—The samples (≃0·5 g) and standards (≃0·1 g) were irradiated in a flux of ≃10^8 n cm^{-2} s^{-1} for 2 h, then set aside for 1 day, and the γ-ray activity due to ^{196}Au was measured at the 355·6-keV photo-peak by means of a Ge(Li) detector and a 1024-channel spectrometer. In the determination of 3·2 to 4·2% of Au, the relative error was <5%; the lower limit of determination was 0·5 mg. CHEM. ABSTR.

1B230. **Analysis of alloys containing platinum, palladium and ruthenium.** Postnikova, I. S. (Baikov Inst. Metall., Acad. Sci. USSR, Moscow). *Zavod. Lab.*, 1977, **43** (6), 641–644 (in Russian).—Procedures involving titration with edta at various pH values and with xylenol orange as indicator are described for determining Pt or Pd in binary or ternary alloys containing lanthanoids, Sc or In. Similar procedures are proposed for the analysis of Pd - V alloys. The sum of Pd and V is determined with edta in the presence of masking agents (depending on the impurities), and the V alone is titrated similarly after the addition of aq. NH_3 to mask Pd. A difference-spectrophotometric procedure, with use of thiourea in 4M-HCl medium and without preliminary separation of Ru, is proposed for the determination of Ru in Ru - Pd - V alloys; the absorbance is measured at 620 nm. Up to 50 mg of V can be tolerated, but oxalic acid, thiourea in excess and ethanol must be added to prevent interference from Pd. A. S. W.

Minerals and non-metallic industrial products

1B231. **Estimation of accuracy of X-ray spectrographic micro-analysis of minerals.** Lavrent'ev, Yu. G.; Pospelova, L. N.; Yurek, K.; and Gulinskii, V. *Tr. Inst. Geol. Geofiz. Akad. Nauk SSR, Sib. Otd.*, 1977, (315), 70–79 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (16), Abstr. No. 16G173.

1B232. **X-ray fluorescence analysis of geological objects with use of a semiconductor detector.** Puravkhaidav, D. (Ob'edin. Inst. Yad. Issled., Dubna, USSR). *Isotopenpraxis*, 1976, **12** (12), 480–484 (in Russian).—The sample (ground to 200 mesh) was dispersed at a surface density of 0·3 to 5 mg cm^{-2} on a polyethylene film covered with a layer of adhesive. The Cu and Mo in Cu - Mo ores were determined simultaneously by measuring their $K\alpha$ lines after irradiation with 22·1-keV radiation from a 15-mCi ^{109}Cd source; the intensity of the lines was measured with a Si(Li) detector (area 30 mm^2; thickness 3 mm; resolution 0·33 keV at 6·4 keV). Matrix effects were avoided by using these 'thin-layer' samples, but a measurement time of 10 to 20 min was needed. By using thicker layers of sample and compensating or accounting for matrix effects, 1% each of Mo and Cu could be determined, after a shorter measurement time, with a relative error of 5%. For W and Nb in W ores, and Sn, Zr and Nb in Sn ores, measurements were made of the $L\alpha$ and

$L\beta$ lines of W, the $K\alpha$ lines of Nb and Zr, and the sum of the $L\alpha$, β and γ radiation of Sn, from 'thin-layer' samples. Only 'thin-layer' samples were suitable for analysis of W ores. CHEM. ABSTR.

1B233. **Use of ion beams for qualitative and quantitative determination of composition of minerals.** Lo Giudice, Antonino; Rimini, Emanuele; Rittmann, Loredana; and Scuderi, Vincenzo (Ist. Mineral., Univ. Catania, Sicily). *Rend. Soc. Ital. Mineral. Petrol.*, 1976, **32** (1), 179–196 (in Italian).—The method described involves bombardment of the sample with 2-MeV He^+ [back-scattering angle 165°; Si(Li) detector] and also with 1·5-MeV H^+ from an ^{241}Am source and measurement of the X-rays produced. Use of He^+ is simple and rapid, and gives good resolution for atomic masses <40; use of H^+ permits unambiguous identification of the elements present. The analysis of olivine and lava samples and the determination of the trace elements Sr, Rb and Zr in lava are reported. L. B.

1B234. **Determination of the isotopic ratio of deuterium to protium at natural abundances of deuterium. I. Isolation of structural water from rock-forming minerals.** Gottstein, Ottomar; and Hejl, Vaclav (Geol. Inst., Acad. Sci., Prague, Czechoslovakia). *Chem. Listy*, 1977, **71** (5), 538–542 (in Czech).—An apparatus is described (with a diagram) in which the sample is fused with V_2O_5 by inductive heating *in vacuo* and the liberated water is trapped at −78° without contamination by, *e.g.*, SO_2 or CO_2. The device has been tested on various rocks containing large amounts of sulphides and carbonates. J. Z.

1B235. **Spectrophotometric determination of calcium, manganese and phosphate in hydroxyapatites.** Rao, S. V. Chiranjeevi (Dept. Chem., G. M. Coll., Sambalpur, India). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 45.—In a soln. of 0·5 g of the mineral, Mn was determined by adding KIO_4, HNO_3, H_3PO_4 and $AgNO_3$, and measuring the absorbance at 526 nm against a sample - reagent soln. in which the MnO_4^- colour had been discharged by a reducing agent. The PO_4^{3-} were determined at 720 nm after addition of $(NH_4)_6Mo_7O_{24}$ and $(NH_4)_2SO_4.FeSO_4$, and Ca was determined at 630 nm after pptn. as its oxalate, dissolution in dil. H_2SO_4, and addition of Eriochrome black T and buffer soln. of pH 11·2. H. L. L.

1B236. **Rapid method for determination of copper in silicates [ceramics and glass] by cathode-ray polarography.** Prasad, R. (Thomas Hill-Jones Ltd., 15 High St., Stratford, London, E.15, England). *Trans. Br. Ceram. Soc.*, 1976, **73**, 5–6.—The sample (<66 μm mesh; 0·1 g) is fused with $LiBO_2$ (0·4 g) in a platinum crucible, the cooled melt is dissolved in 5·3% HNO_3 (10 ml) during 1 h, and the clear soln. is diluted to 50 ml with dil. HNO_3 after addition of tartaric acid (0·5 g). A 2-ml aliquot is then subjected to single-sweep polarography (dropping-mercury electrode and silver - AgCl reference electrode) between 0 and −0·5 V, and the recorded peak-current at $\simeq -0{\cdot}3$ V is referred to a calibration graph. Results for 0·03 to 1% of Cu in samples from a cup, a tile, pottery and glass showed similar accuracy and better reproducibility compared with those obtained by a.a.s. There is interference from a >10-fold excess of Tl or Pb, but Cd can be tolerated. W. J. B.

1B237. **Quantitative analysis of thin films by X-ray fluorescence and diffractometry.** Eberspaecher, O.; and Hieber, K. (Forschungslab., Siemens A.-G., Muenchen, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 567–574 (in German).—Thicknesses of films of Ag and Mo on glass and on silicon single crystals in the range 3 to 200 nm were determined by X-ray fluorescence analysis with use of an energy-dispersive detector; the detection limits were 70 ng cm^{-2} for Mo and 215 ng cm^{-2} for Ag. J. W. P.

1B238. **Analysis of anodic oxide films on gallium arsenide and gallium phosphide by means of radioactive tracer techniques.** Verplanke, J. C.; and Tijburg, R. P. (Philips Research Laboratories, Eindhoven, Netherlands). *J. Electrochem. Soc.*, 1977, **124** (5), 802–804.—Samples were irradiated in a thermal-neutron flux of $\simeq 3 \times 10^{11}$ n cm^{-2} s^{-1} for 2 h before anodisation in ethanediol - 3% citric acid (pH adjusted to 6 with NH_3) (2:1). The oxide layer was then removed with 10% H_3PO_4 at 80°. The activities of ^{72}Ga and ^{76}As (from GaAs) in the anodisation and etching baths were measured with a Ge(Li) detector and a 2048-channel analyser; the ^{72}Ga activity from GaP was measured with a NaI(Tl) detector and a 400-channel analyser, and the ^{32}P activity with a liquid scintillation counter after the decay of ^{72}Ga. Ratios of Ga to As found in oxide layers after annealing GaAs at temp. between 200° and 600° are reported. Besides the average composition of the films, the composition of anodic oxide layers on GaAs as a function of thickness was determined. Dependence of the oxide layer composition on the nature of the electrolyte is discussed. J. H. A. R.

1B239. **Spectrophotometric determination of vanadium(IV) in catalysts.** Novoselova, I. M.; and Barkovskii, V. F. (Ural Chem. Sci.-Res. Inst., Sverdlovsk, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1247–1249 (in Russian).—Quadrivalent V forms a red - violet heteropoly-anion with WO_4^{2-} and PO_4^{3-} in an aq. medium 0·25M in H_3PO_4, 0·25M in H_2SO_4 and 0·08M in Na_2WO_4; the reaction mixture should be heated at 100° for 10 min. The complex exhibits max. absorption at 540 nm, and Beer's law is obeyed for 0·5 to 6 μg ml^{-1} of V^{IV}; 5-fold amounts of V^V, Fe^{III} or Cr^{III} do not interfere. To determine V^{IV} in a V^{IV} - V^V catalyst, the sample (0·2 g) is initially decomposed by hot 40% H_3PO_4 (10 ml) and conc. HF (3 ml). M. P.

1B240. **Analysis of palladium - lead catalysts.** Kurakina, T. V.; Sokolova, A. G.; and Popova, N. M. (Vses. Nauchno-Issled. Inst. Sint. Nat. Dushistykh Veshchestv, Kaluzhskoye, USSR). *Maslob.-Zhir. Prom-st.*, 1976, (11), 30–32 (in Russian).—Such a catalyst (with a $CaCO_3$ carrier), used in the synthesis of linalyl acetate, was analysed by complexometric titrimetry. A 0·5-g sample was dissolved in dil. HCl - HNO_3, and a portion of the diluted soln. was treated with excess of Na_2edta; the total Pd and Pb was then determined by back-titration at pH 6 with 0·05N-Zn acetate (xylenol orange as indicator). Lead was pptd., as Pb(OH)Cl, in another aliquot by treatment with aq. NH_3 - NH_4Cl at 80°, and the ppt. was filtered off. The diluted filtrate was adjusted to pH 10 with aq. NH_3 buffer, and Ca was determined in the alkaline soln. by titration with edta, with Acid chrome dark blue (C. I. Mordant Blue 13) as indicator. The Pb(OH)Cl ppt. was dissolved in acetate buffer soln. (pH 6), and Pb was determined by back-titration with Zn

acetate soln. after addition of excess of edta. The analysis time was ≃2 h, and the relative error was generally <7%. CHEM. ABSTR.

1B241. **Determination of platinum and iridium in reforming catalysts by flameless atomic-absorption spectrometry.** Tittarelli, P. (Stn. Sper. Combust., San Donato Milanese, Italy). *Riv. Combust.*, 1976, **30** (9–10), 289–292 (in Italian).—The sample (typically containing 0·3 to 0·4% of Pt and 0·03 to 0·07% of Ir) was treated with H_3PO_4 to dissolve Al_2O_3, then the Pt and Ir were dissolved in aqua regia. This soln. was subsequently transferred to a graphite-tube furnace for drying at 110°, ashing at 1800° (for Pt) or 1300° (for Ir) and atomisation at 2750°; the absorption was measured at 265·9 nm for Pt and 264 nm for Ir. Results compared satisfactorily with those obtained by X-ray fluorescence, but, for Ir, matrix interference could not be completely removed. CHEM. ABSTR.

1B242. **Determination of melment [lignin sulphonate] in cement mixtures by ultra-violet spectrophotometry.** Reul, H. (Zentrallab., Chemische Fabrik Gruenau, Illertissen, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **285** (2), 126 (in German).—The melment is extracted by treatment of the sample with 10% NaOH soln. The soln. is separated by centrifugation and neutralised with M-HCl, and the absorbance of the diluted soln. is measured at 220 nm. The accuracy is within ±1·5% for contents >20% and within ±3% for those <1%. D. B. A.

See also Abstracts: **1A1**, Determination of impurity elements in inorganic samples: review. **1A2**, Flame-photometric determination of Na and K. **1A3**, Various reagents for inorganic species: review. **1A4**, Reagents for metals and anions: review. **1A5**, Reagent for various metals. **1A6**, Reagents for Cu and precious metals. **1A8**, Oxidising titrant for various inorganic species. **1C13**, Analysis of complexes of Ni and Zn. **1C49**, Polarography of complexes of Zn and Cd. **1C98**, Determination of Pb in wax crayons. **1D14**, Determination of Ba. **1D15**, Determination of Cd. **1E9**, Determination of Fe and Co in drugs. **1F8**, Determination of NO_3^-. **1H9**, Determination of HCN in fire gases. **1H24**, Determination of traces of metal ions. **1H28**, Determination of U. **1H31**, Determination of In. **1H33**, Determination of C. **1H38**, **1H39**, Determination of As in minerals. **1H55**, **1J76**. Determination of Hg. **1J5**, Determination of O in gases. **1J8**, Measurement of gases in nuclear fuel, nuclear waste and alloys. **1J13**, Materials for collection of trace metals. **1J19**, Detector for inorganic S compounds. **1J20** to **1J22**, Inorganic ion exchangers. **1J40**, Detector selective for Br. **1J41**, Detector selective for Si. **1J50**, Micro-probe analysis of steels. **1J53**, X-ray valency band spectra of Cu and its oxides. **1J55** to **1J58**, X-ray fluorescence analysis of inorganic samples. **1J61**, Background correction in micro-probe analysis of metals and alloys. **1J63**, High-purity water for a.a.s. **1J64**, Lamps for u.v. spectrometry of elements. **1J65**, Furnace tubes for a.a.s. of metals. **1J66**, Determination of Zn. **1J67**, Determination of Pb, Sn and Zn in minerals. **1J68**, Spectrographic analysis for metals. **1J69**, Improvements in spectroscopy of metals. **1J81**, Flameless a.a.s. of elements. **1J91**, **1J92**, Auger-electron spectroscopy of metal surfaces. **1J93**, X-ray photoelectron spectroscopy of oxide films. **1J94**, **1J95**, **1J97**, **1J98**, Inorganic applications of m.s. **1J99**, Determinations with use of two radionuclides: review. **1J103**, Measurement of radioactive noble gases. **1J104**, Electrochemical techniques for determination of metals: review. **1J106**, E.m.f. values for mineral acids. **1J107**, Electrode responsive to Ag^+. **1J108**, Meter for measurement of F^-. **1J109**, Electro-deposition of Cd, Cu and Hg. **1J111**, Anodic-stripping voltammetry of Cd, Cu, Pb and Zn.

C—ORGANIC CHEMISTRY

General

1C1. **Gel-permeation chromatography of low-molecular-weight materials with high-efficiency columns.** Krishen, Anoop; and Tucker, Ralph G. (The Goodyear Tire & Rubber Co., Res. Div., Akron, Ohio 44316, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 898–902.—Rapid separations of, *e.g.*, n-alkanes; aromatic and cyclic hydrocarbons; mono- and poly-hydric alcohols; esters; phenols and amines; condensation products (nonylphenol - formaldehyde); oligomers of 1,2-dihydro-2,2,4-trimethylquinoline; and accelerators or plasticisers (mol. wt. 100 to 2000) were achieved by injecting 1 to 5 μl of a soln. of the sample (60 to 70 mg ml^{-1}) in tetrahydrofuran on to a stainless-steel column (610 mm × 8 mm) packed with a styrene - divinylbenzene copolymer gel. Elution was effected with tetrahydrofuran (at 0·5 ml min^{-1}), a differential refractive-index detector and a dual-beam u.v. detector (254 nm) were used to monitor the eluate, and a single pump provided reference and analytical flows without stream splitting. Advantages of the technique and some of its limitations are discussed. W. C. D.

1C2. **Separation of complex [organic] mixtures by multi-stage preparative gas chromatography.** Fedchenko, G. S.; and Vigdergauz, M. S. *Khim. Tekhnol. Topl. Masel*, 1977, (3), 61–63 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (16), Abstr. No. 16G235.

1C3. **Coulometric determination of small amounts of water in aprotic polar solvents.** Bykova, L. N.; Petrov, S. I.; Khusainov, M. G.; Bogoslovskii, V. V.; and Petrova, T. N. (Moscow Textile Inst., USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 938–944 (in Russian).—From 0·05 to 1% of H_2O in dimethylformamide, dimethylacetamide or dimethyl sulphoxide has been determined by using electro-generated iodine in Karl Fischer reagent as titrant. The iodine was generated at a platinum anode operated *vs.* a platinum cathode at a c.d. of 5 mA cm^{-2}. The end-point was indicated amperometrically by using two platinum electrodes kept at a p.d. of 100 mV, the iodine being generated, after adding the sample, to reach the initial indication-current value (60 μA). Up to 60% (v/v) of dimethyl-formamide or -acetamide in the reaction mixture has no effect on the accuracy and reproducibility. The concn. of dimethyl sulphoxide in the reaction mixture must be constant or <10% otherwise results are erroneous. M. P.

Elements and functional groups

1C4. **Chromatographic determination of relative retention of isotopic species of oxygen in methanol and [*methyl*-$^2H_3$]methanol.** Pauls, R. E.; Mahle,

N. H.; Shepard, A. T.; Gaw, Jenny Chow; and Rogers, L. B. (Dept. Chem., Univ. Georgia, Athens, Ga. 30602, U.S.A.). *Sep. Sci.*, 1977, **12** (3), 307–320.—Separations were effected by g.c. on (*i*) a stainless-steel column (4 m × 2·2 mm) packed with 14% of glycerol on AW-DMCS Chromosorb W (100 to 120 mesh), or (*ii*) a column (2·7 m × 2·2 mm) of Porapak T (100 to 120 mesh); N was used as carrier gas (0·3 ml min^{-1}). The column effluents were monitored by a computer-operated quadrupole mass spectrometer (operated in the multiple-ion mode) interfaced with an on-line PDP-11/20 data-acquisition system. Relative retention values and differential thermodynamic data are reported. M. J. SL.

1C5. **Improvement of oxygen determination method in organic compounds through addition of chlorohydrocarbon vapour to carrier gas.** Kirsten, Wolfgang J. (Dept. Chem., Agric. Coll. Sweden, Uppsala). *Microchem. J.*, 1977, **22** (1), 60–64.—The modifications to methods of O determination with a Carlo Erba automatic elemental analyser are described. The stainless-steel separation column was replaced by a glass one (23 cm × 4 mm) and a loosely compressed layer of nickel wire was introduced into the pyrolysis tube to prevent clogging by decomposition of the 1-chloropentane that was volatilised into the carrier gas (30 or 50 ml min^{-1}) at the rate of 18 $\mu l\ h^{-1}$. The results for 12 samples (≃0·5 mg), including 3,5-dinitrobenzoic acid, sucrose and $AgNO_3$, showed good agreement with calculated values. T. G. A.

1C6. **Micro-determination of oxygen at trace levels in organic sulphur compounds.** Bozier, C.; Grall, M.; Fraisse, D.; and Muller, C. (Soc. Nationale Elf-Aquitaine, Lacq, France). *Microchem. J.*, 1977, **22** (2), 249–258.—The method consists in pyrolysis of the sample in N, reduction of the gases over amorphous carbon at 1120° and purification of the resulting CO for determination in an i.r. cell (path-length 25 cm). The calibration graph is rectilinear for up to ≃400 μg of O, and the accuracy is much higher than that of other methods when O contents are low, *e.g.*, <1% in polymers. W. J. B.

1C7. **Determination of fluorine in [organic] tetrafluoroborates.** Volodina, M. A.; Gorshkova, T. A.; and Morozova, O. P. *Vestn. Mosk. Univ., Khim.*, 1977, **18** (2), 230–233 (in Russian).—The complex (2·5 to 5·0 mg) is mixed with ≃200 mg of $(NH_4)_2SO_4$ in a silica tube and heated in a stream of NH_3 (3 to 5 ml min^{-1}) for 10 to 15 min at 800° to 850°. The liberated NH_4F is dissolved in H_2O and the F^- are determined by thorimetric titration. The method has been applied to determination of F in tetraphenylbismuthonium tetrafluoroborate, the Bi being also determined gravimetrically as Bi_2O_3 after the decomposition. M. R. M.

1C8. **Ultra-micro determination of nitrogen in organic compounds. VIII. Weighing of hygroscopic and volatile samples for the sealed-tube method.** Miyahara, Keikichi; and Takaoka, Tomo (Res. Lab., Shionogi and Co. Ltd., Osaka, Japan). *Microchem. J.*, 1977, **22** (1), 7–14.—Copper foil (2 cm × 2 cm and 0·15 to 0·2 mm thick) was used in the construction of capsules (with folded ends) that were suitable for drying 0·3 to 0·5 mg of such samples *in vacuo* for subsequent weighing and determination of N by the sealed-tube method (*cf.* Kirsten and Hozumi, *Anal. Abstr.*, 1963, **10**, 4211). The chemical treatment of the capsules to ensure total combustion is described. Results for nine compounds, including cephalexin and pyrazine, are given. (For Part VII see *Anal. Abstr.*, 1977, **33**, 1D22.) T. G. A.

1C9. **Ultra-micro determination of nitrogen in organic compounds. IX. New method for handling volatile liquids in the sealed-tube method.** Miyahara, Keikichi; and Takaoka, Tomo (Res. Lab., Shionogi and Co. Ltd., Osaka, Japan). *Microchem. J.*, 1977, **22** (2), 210–215.—The liquid (300 to 600 μg) is drawn into a glass capillary tube (≃2 cm × 0·5 mm o.d.) and placed in a glass weighing tube (35 mm × 0·75 mm o.d.; wall thickness ≃0·1 mm). Before sealing the weighing tube, the air is replaced by a mixture of CO_2 and CH_4 having approx. the same density as air. This tube (slightly scratched) and some copper gauze are placed at the bottom of the O-filled combustion tube, which is then sealed; the weighing tube is broken by tapping before combustion. Results for N in nitrobenzene, nitroethane and other volatile liquids confirm the accuracy of the method. (For Part VIII see preceding abstract.) W. J. B.

1C10. **Ultra-micro determination of nitrogen in organic compounds. X. Handling volatile solid samples in the sealed-tube method.** Miyahara, Keikichi; and Takaoka, Tomo (Res. Lab., Shionogi and Co. Ltd., Osaka, Japan). *Microchem. J.*, 1977, **22** (2), 216–221.—The procedure used for volatile liquids (see preceding abstract) is slightly modified by placing the solid directly in the bottom of the weighing tube (o.d. 1·5 mm), and then constricting the tube (to ≃0·5 mm i.d.) ≃1·5 cm from the open end. Replacement of the air with CO_2 - CH_4, sealing and weighing are as described previously. Results for pyrazine, sulphathiazole and three other solids confirm the validity of the method and that no correction is necessary for wt. changes after replacement of air. W. J. B.

1C11. **Simultaneous determination of phosphorus, chlorine and bromine in organophosphorus compounds by using activation with fast neutrons.** Lisovskii, I. P.; and Smakhtin, L. A. (L. Ya. Karpov Phys.-Chem. Inst., Obninsk Branch, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1214–1216 (in Russian).—The sample (a mixed chloro- and bromo-alkyl derivative of phosphoric or phosphinic acid), in a steel container, was irradiated in a flux of ≃2 × 10^{10} n s^{-1} for 1·5 min. To determine P and Br, the γ-ray activities of ^{28}Al and ^{78}Br were measured at 1·78 MeV and 0·51 MeV, respectively, with a NaI(Tl) detector. To determine Cl, the γ-ray activity of ^{37}S at 3·1 MeV was measured with a well-type NaI(Tl) detector. The coeff. of variation were <5% when determining ≃8% of P, 9·7 to 20% of Cl and 43 to 47% of Br. Correction coeff. were used to eliminate inter-isotope effects. M. P.

1C12. **Ion[-exchange] chromatographic determination of sulphur and chlorine using milligram and sub-milligram sample weights.** Smith, F., jun.; McMurtrie, A.; and Galbraith, H. (Dionex Corp., 1228 Titan Way, Sunnyvale, Calif. 94086, U.S.A.). *Microchem. J.*, 1977, **22** (1), 45–49.—The samples (50 μg to 150 mg) with Cl contents 0·4 to 50% and S contents 0·015 to 33% were burnt in a Schoniger flask and the products were absorbed in 3% H_2O_2 soln. - 3mM-$NaHCO_3$ - 2·4mM-Na_2CO_3. The soln. was then analysed with use of a Dionex model 10 ion chromatograph with a low-capacity anion-exchange separator resin and a high-capacity cation-exchange suppressor resin, 3mM-$NaHCO_3$ - 2·4mM-

Na_2CO_3 as eluent and conductivity detection. Calibration graphs were rectilinear for 0·25 to 10 mg of *S*-benzylthiouronium chloride (17·49% of Cl, 15·82% of S). Results for Cl and S are compared with those obtained by argentimetric titration and pptn. as $BaSO_4$, respectively. (*Cf.* Small *et al.*, *Anal. Abstr.*, 1976, **30**, 5J17.) T. G. A.

1C13. **Micro-analysis of metal complexes by atomic-absorption spectrophotometry.** Davydova, S. L.; Domanina, O. N.; Voronina, Z. D.; and Rapoport, L. M. (A. V. Topchiev Inst. Petrochem. Synth., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (6), 1171–1175 (in Russian).—This technique has been used to determine the metal component in complexes of Ni and of Zn with nine Schiff bases [including *N*-(2-hydroxybenzylidene)aniline, 5-ethyl-3-(phenyliminomethyl)thiophen-2-thiol and *N*-(α-phenylimino-*o*-tolyl)toluenesulphonamide]. The sample (2 mg) was dissolved in dimethylformamide to give a soln. containing ≃0·5 mg l^{-1} of Zn and/or ≃2 to 3 mg l^{-1} of Ni. This soln. was aspirated directly into an air - acetylene flame and the absorption was measured at 232·2 nm for Ni and 213·7 nm for Zn. The coeff. of variation was <1% (10 to 12 results) when determining ≃8 to 19% of Zn and 9 to 15% of Ni. The results agreed with values calculated from the stoicheiometry of the chelates. M. P.

Compounds

1C14. **Diffusion and partition of n-alkanes in dimethylsilicone stationary phases.** Millen, William; and Hawkes, Stephen (Dept. Chem., Oregon State Univ., Corvallis, Ore. 97331, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (5), 148–150.—Diffusion of linear alkanes (up to C_{11}) is faster in the polydimethylsilicone elastomer SE-30 than in the oil SF-96-2000, and the reverse occurs for alkanes longer than C_{13}. Equations are derived for determining partition coeff., entropy and enthalpy for evaporation of alkanes from SE-30. Symmetrical peaks were obtained at temp. as low as 50° on SE-30 (0·291% on 60 to 80-mesh glass beads). M. J. St.

1C15. **[Spectrophotometric] determination of carbonyl compounds [in hydrocarbons], as hydrazones.** Kuznetsova, E. V.; Taranenko, S. A.; Mil'kina, T. N.; and Batalin, O. E. (All-Union Sci.-Res. Inst. Petrochem. Processes, Leningrad, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1185–1189 (in Russian).—To determine traces of aldehydes and ketones in, *e.g.*, isoprene, pentane, hexane, benzene or cyclohexane, the sample (10 ml) was mixed with satd. 2,4-dinitrophenylhydrazine (**I**) soln. in benzene (10 ml) and 1% H_3PO_4 soln. in heptanol (0·5 ml). The mixture was set aside at ambient temp. for 1 h, and the unreacted **I** and H_3PO_4 were extracted with H_2O (3 × 100 ml). The organic phase was concentrated, then treated with ethanolic 20% KOH (5 ml) and diluted with ethanol to 25 ml, and the absorbance of the soln. was measured with use of a blue filter. The relative error was <±11% when determining 0·4 to 121 p.p.m. of acetone, isovaleraldehyde, methacrylaldehyde and/or acetaldehyde. M. P.

1C16. **Simple spectrophotometric determination of primary, secondary and tertiary alcohols.** Leenson, I. A.; and Sergeev, G. B. (M. V. Lomonosov Moscow State Univ., USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 1016–1019 (in Russian).—The alcohol sample (several drops) is mixed with 6M-HCl (5 ml), then hexane (1 ml) and aq. 20% $NaNO_2$ (2 ml) are added, and the mixture is shaken to convert the alcohols into alkyl nitrites. The organic phase (several drops) is then diluted with hexane, and the type of alcohol is identified from the shape of the absorption spectrum in the range 340 to 410 nm. The nitrites corresponding to tertiary alcohols are identified by characteristic absorption at ≃400 nm. Those from both primary and secondary alcohols have absorption max. at ≃358 nm and 345 nm, and a parameter [α, the ratio of the height of the max. at 358 nm to that of the max. at 345 nm (measured relative to the minimum between those two maxima)] is used to distinguish between them. The nitrites from primary alcohols have α values of ≃1·3 to 1·35 (except that from methanol, for which α = 0·98); nitrites from secondary alcohols have α values of ≃3·1 to 3·6. Binary mixtures of primary and secondary alcohols containing >10% of the primary alcohol can be analysed by using a calibration graph relating the α value to the concn. of the primary alcohol in the mixture. The method was most useful for C_3 to C_7 alcohols. M. P.

1C17. **Group identification of organic compounds by thin-layer chromatography.** Sliwiok, Jozsef; and Wira, Halina (Inst. Chem., Silesian Univ., Katowice, Poland). *Microchem. J.*, 1977, **22** (2), 135–143.—Group identification of homologous compounds is based on the constant differences ΔR_F and ΔR_M as calculated from rectilinear graphs constructed from data obtained by t.l.c. on Kieselguhr G impregnated with a 15% soln. of liquid paraffin in benzene with use of two mobile phases [acetic acid - liquid paraffin - H_2O (8 : 1 : 1, and 7 : 2 : 1)]. Separation of groups was also possible from the colours and stabilities of the spots revealed with aq. thymolphthalein and with molybdophosphoric acid. The method was applied to six higher (up to C_{20}) aliphatic alcohols and the corresponding acids and ethyl esters. W. J. B.

1C18. **Quantitative head-space gas analysis by standard-additions method. Determination of hydrophilic solutes in equilibrated gas - aqueous liquid systems.** Drozd, Josef; and Novak, Josef (Inst. Anal. Chem., Czech. Acad. Sci., Brno). *J. Chromatogr.*, 1977, **136** (1), 37–44.—Aqueous soln. of methanol, ethanol, propanol and acetone are analysed by injecting equilibrated head-space gases (2 ml) on to a column (18 cm × 3 mm) packed with Porapak P (80 to 100 mesh) and operated at 120° or 130°, with N as carrier gas and a flame ionisation detector. At solute concn. of 1 to 100 p.p.m. the error was ≃20 to 3%. The sensitivity to acetone and propanol can be improved by saturating the liquid phase with Na_2CO_3. M. J. St.

1C19. **Variable-time kinetic determination of isopropyl alcohol and other alcohols by means of mutual induction in oxidations by chromium(VI).** Veres, Sandor; and Csanyi, Laszlo J. (Reaction Kinetics Res. Gr., Hungarian Acad. Sci., Attila Jozsef Univ., Szeged). *Anal. Chem.*, 1977, **49** (7), 980–983.—The method is based on oxidation of the alcohols by chromic acid, the reaction being accelerated by the addition of oxalic acid. The reaction time is measured from the moment of mixing, and the absorbance of the soln. is measured at 350 nm either at periodic intervals or with continuous recording. The coeff. of variation is <5% (4 or 5 replicate measurements at 9 different concn. levels from mM to 0·4M). G. P. C.

1C20. **Reactivity of monosaccharides and their derivatives with aromatic amines. I. Preliminary study.** Desmaison, [Mrs.] A. M.; and Beneytout, J. L. (Lab. Chim. Biol., Fac. Med. Pharm., Limoges, France). *Bull. Soc. Pharm. Bordeaux*, 1976, **115** (2), 69–74 (in French).—Colour reactions between monosaccharides (and their derivatives) and aromatic amines have been studied for the purpose of detecting the sugars or their derivatives. A mixture of test soln. (1 ml) and amine soln. (5 ml) was heated at 100° for 10 min, then cooled to room temp., and the absorption was examined over the range 350 to 700 nm. Aldohexoses (*e.g.*, glucose and galactose) and disaccharides (*e.g.*, lactose and sucrose) formed green chromophores (max. absorption at ≃620 nm) with *ortho*-substituted or alkylated amines. This reaction was specific, no colour being produced by ketohexoses or pentoses. Aldopentoses and glucuronic acid reacted with 2,6-xylidine to form a pink chromophore (max. absorption at 520 nm); hexoses did not react with this amine.

CHEM. ABSTR.

1C21. **Fully automatic ion-exchange and gel-permeation chromatography of neutral monosaccharides and oligosaccharides with a JEOL JLC-6AH analyser.** Kennedy, John F.; and Fox, John E. (Macromol. Serv., Dept. Chem., Univ. Birmingham, England). *Carbohydr. Res.*, 1977, **54** (1), 13–21.—This amino-acid analyser was modified by installation of (*a*) a piston pump suitable for conc. H_2SO_4, (*b*) absorbance filters for measurements at 425 and 510 nm, and (*c*) a second duplicate column. For ion-exchange separation, the columns were packed with LC-R-3 resin (JEOL) and were programmed for elution of mono- and oligo-saccharides with 0·13 to 0·5M-$K_2B_4O_7$ at 55° in the descending mode and assay with orcinol - H_2SO_4 reagent; the absorbance was monitored at 425 nm (corrected for absorbance at 510 nm). For gel-permeation chromatography, the columns were packed with Bio-Gel P2 (400 mesh), and the sugars were eluted with de-aerated H_2O (saturated with toluene) at 65° in the ascending mode; the same colorimetric reagent was used. The respective analysis times were 6·5 h and 20 to 22 h for the two methods cited. A typical profile of a starch hydrolysate shows the separation of carbohydrates on the basis of molecular size. R. F. S.

1C22. **Gas-chromatographic analysis of non-esterified lower sugars and polyhydric alcohols.** Korol', A. N. (Inst. Phys. Chem., Acad. Sci. Ukr. SSR, Kiev). *Zh. Anal. Khim.*, 1977, **32** (5), 1020–1024 (in Russian).—Thirteen products of the condensation of formaldehyde at 80° in aq. soln. containing a Pb catalyst were separated by g.c. on a column (3 m × 3 mm) of Polysorb 4, operated with temp.-programming from 170° to 245° at 3° min^{-1}, He (30 ml min^{-1}) as carrier gas and thermal-conductivity detection. The products identified included glycollaldehyde, ethanediol, glyceraldehyde, dihydroxyacetone, glycerol and erythrose (down to 0·1% of the sugars could be determined by using 4-methylpentanol as internal standard). M. P.

1C23. **Quantitative determination of D-xylose by a coupled reaction of D-xylose isomerase with D-glucitol dehydrogenase.** Kersters-Hilderson, Hilda; Van Doorslaer, Elisa; De Bruyne, Clement K.; and Yamanaka, Kei (Lab. Algemene en Biol. Scheikunde, Rijksuniv., Gent, Belgium). *Anal. Biochem.*, 1977, **80** (1), 41–50.—The D-xylose soln. (0·5 ml) was added to 0·017M-maleate buffer of pH 6 (0·4 ml) followed by 0·033M-$MnCl_2$ (50 μl), 0·1M-glutathione (10 μl), 0·015M-NADH (10 μl), D-glucitol dehydrogenase (50 μl, containing 4·5 i.u.) and D-xylose isomerase (10 μl, containing 1 i.u.). The decrease in NADH concn. (at 30°) was monitored at 340 nm, and the readings were referred to a calibration graph for D-xylose. The results obtained were reproducible and agreed well with those of the *o*-toluidine - acetic acid method. The present method was also used for the assay of exo-1,4-β-xylosidase (from *Bacillus pumilus*) with use of xylobiose as substrate, the α-D-xylose formed being determined as stated. R. A. H.

1C24. **Chromatographic 'spectra' of ketones.** Satcs, V.; Avots, A.; and Belikov, V. A. *Latv. PSR Zinat. Akad. Vestis, Kim. Ser.*, 1977, (1), 64–68 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (16), Abstr. No. 16G248.

1C25. **Identification of impurities in acetone by gas - liquid chromatography.** Bruk, A. Yu.; Gaishun, K. A.; and Markova, V. A. (Grozny Chem. Plant, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1200–1205 (in Russian).—Twenty-two compounds (hydrocarbons, ketones, ethers, esters, alcohols, carboxylic acids and aldehydes) were identified in technical-grade acetone by means of their retention values obtained with use of a column (1 m × 2·5 mm) packed with 30% of polyoxyethylene glycol 300 on Chromosorb R and a column (8 m × 3 mm) packed with 2·45% of glycerol plus 4·8% of 1,2,3-tris-(2-cyanoethoxy)propane on INZ-600, each operated at 50° with N as carrier gas and flame ionisation detection, and a column (1·3 m × 2·5 mm) packed with Polysorb-1 and operated at 130°, with H as carrier gas and thermal-conductivity detection. M. P.

1C26. **Gas-chromatographic determination of formic and acetic acids and formaldehyde in aqueous medium.** Sizova, G. S.; Usova, E. P.; and Znamenskaya, A. P. (State Sci.-Res. and Design Inst. Nitrogen Ind. and Org. Synth. Products, Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 1034–1036 (in Russian).—These compounds (by-products in the manufacture of benzoic acid by oxidation of toluene) are determined by successive treatment of the aq. reaction mixture (1·5 ml) with ethanol (3·5 ml), ethanolic 0·2% toluene-*p*-sulphonic acid (1 ml) and ethanolic 1% ethyl methyl ketone (0·5 ml) as internal standard, and the mixture is set aside at ambient temp. for 30 min to reach esterification equilibrium. Ethyl formate, ethyl acetate and diethoxymethane formed are separated and determined by g.l.c. at 65° on a column (2 m × 3 mm) packed with 20% of polyoxyethylene glycol 400 on Chromaton N AW-DMCS, with N (15 ml min^{-1}) as carrier gas and flame ionisation detection. The coeff. of variation was <8% for ≃3 to 15 mg ml^{-1} of the compounds cited. M. P.

1C27. **Application of open-tubular gas - liquid chromatography in an investigation of the stability of pristanic and phytanic acids towards alkaline treatment of their methyl or (−)-menthyl esters.** Kates, M.; Hancock, A. J.; and Ackman, R. G. (Dept. Biochem., Univ., Ottawa, Ont., Canada). *J. Chromatogr. Sci.*, 1977, **15** (5), 177–180.—Gas chromatography at 170° on a column (46 m × 0·25 mm) coated with butanediol succinate and with He as carrier gas (4 ml min^{-1}) gives effective separation of 2*S*,6*R*,10*R*- and 2*R*,6*R*,10*R*-isomers of (−)-menthyl pristanate, but inferior separation of the corresponding methyl esters; however, a column 92 m long will resolve a mixture of the methyl esters containing 2% of the 2*S*,6*R*,10*R*-isomer. No

epimerisation at the C atom in position 2 of pristanic acid or its methyl or (−)-menthyl ester is detected as a result of treatment with aq. or methanolic M-NbOH (under reflux for 1 h or at 120° in a sealed tuae for 24 h), nor at the C atom in position 3 of phytanic acid or its methyl ester. M. J. SL.

1C28. **Polarographic behaviour of saturated dicarboxylic acids in acetone.** Kreshkov, A. P.; Rogatinskaya, S. L.; and Emirdzhanova, G. N. (D. I. Mendeleev Moscow Chem.-Technol. Inst., USSR). *Zh. Obshch. Khim.*, 1977, **47** (6), 1379–1383 (in Russian).—In the polarography of oxalic, malonic, succinic, glutaric and adipic acids in acetone containing 0·1M-tetraethylammonium perchlorate as supporting electrolyte, the $E_{\frac{1}{2}}$ values (V) and corresponding concn. (mM) were: −1·45 and −1·58, 2 and 5; −1·52 and −1·74, 0·8 and 4; −1·76 and −1·90, 1 and 5; −1·85 and −1·90, 1 and 4; and −1·87 and −2·04, 1 and 8. The polarograms each show one well-defined wave, and reduction is irreversible. Acetone acts as a differentiating solvent on acids that have closely similar strengths in aq. soln., thus affording a means of determining such acids in their mixtures. V. S.

1C29. **Fatty acids. XV. Thin-layer chromatographic behaviour of acetylenic fatty esters on silicic acid.** Jie, M. S. F. Lie Ken; and Lam, C. H. (Chem. Dept., Univ. Hong Kong). *J. Chromatogr.*, 1977, **136** (1), 178–181.—The methyl esters (20 to 30 μg in 1 μl of acetone) were subjected to t.l.c. on glass plates coated with Silica gel 60 GF_{254} and activated at 120° for 1·5 h. The chromatograms were developed with light petroleum (boiling-range 60° to 80°) - ethyl ether (23 : 3), and the compounds were located by spraying with ethanolic 10% molybdophosphoric acid and heating at 150° to 200° for ≃10 min (or until the spots became visible). The R_F values of 19 esters are reported. M. J. SL.

1C30. **Cyano-group-containing stationary phases for chromatographic study of products of the catalytic synthesis of nitriles.** Khusainov, Kh. Sh.; and Zakirov, N. S. (Samarkand. Gos. Univ. im Navoi, Uzb. SSR). *Dokl. Akad. Nauk Uzb. SSR*, 1976, (6), 55–56 (in Russian).—Compounds containing one, two or three cyano-groups were tested for this purpose. The log. of the relative retention time was a rectilinear function of C number for the homologous series of alcohols, aldehydes, nitriles, amines and hydrocarbons that were used to evaluate the stationary phases. The best compounds for the purpose were 1,2-bis-(2-cyanoethoxy)ethane, 3,3′-thiodipropionitrile and 1,2,3-tris-(2-cyanoethoxy)-propane, which could be used at temp. up to 120°, 100° and 160°, respectively. CHEM. ABSTR.

1C31. **Separation and determination of some amines by ion-exchange chromatography.** Rawat, J. P.; and Singh, J. P. (Dept. Chem., Aligarh Muslim Univ., U.P., India). *Sep. Sci.*, 1977, **12** (3), 281–288.—The hydrochlorides of 15 amines (including, *e.g.*, dimethylamine, aniline, pyridine and isoquinoline) were separated on Whatman No. 3 paper strips (15 cm × 2·5 cm), impregnated with tin^{IV} molybdate soln. in various concn. of aq. $NaNO_3$ and HCl, by development with M-HCl or mM-$NaNO_3$; the spots were detected with 0·2% ninhydrin soln. in butanol (saturated with H_2O) or 1% 4-dimethylaminobenzaldehyde soln. in ethanol - HCl (19 : 1). Graphs of pH *vs.* R_M values are presented for 12 of the amines. Separations were also effected on a glass column containing 2 g of tin^{IV} molybdate (supported on glass wool) by elution with H_2O and or aq. 1% $Fe(NO_3)_3$; 10-ml fractions of the eluate were analysed by spectrophotometry. M. J. SL.

1C32. **Conductometric titration of amines with edta in dimethyl sulphoxide.** Yoshimura, Chozo; and Ichihara, Haruo (Dept. Appl. Chem., Fac. Sci. and Eng., Kinki Univ., Higashi-Osaka-shi, Osaka, Japan). *Bunseki Kagaku*, 1977, **26** (6), 375–379 (in Japanese).—The titration of dimethylamine, ethylamine, diethylamine, aniline, *NN*-dimethylaniline, diphenylamine and 4-aminobenzoic acid was studied. A sharp inflection point was observed for the titration of the aliphatic amines with edta at a molar ratio of 4 : 1. No inflection point was observed for the titration of the aromatic amines. The aliphatic amines were titrated successfully in the presence of DL-serine, DL-methionine and DL-threonine. The hydrochlorides of L-glutamic acid, glycine, L-cystine and L-aspartic acid also showed inflection points at the 4 : 1 molar ratio with edta.

1C33. **Titration of 1-alkyl-1-nitrosohydrazines with iron(III).** Lanovaya, G. A.; Fedorova, V. A.; and Beresneva, V. I. (Siberian Technol. Inst., Krasnoyarsk, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1206–1209 (in Russian).—To determine the ethyl, propyl, butyl, pentyl or hydroxyethyl derivative of nitrosohydrazine, the test soln. (10 ml, containing 0·01 to 0·03 g of the compound) was made 0·1N in $Fe_2(SO_4)_3$, the resulting violet soln. was heated at 100° for ≃1 min to reduce Fe^{III} to Fe^{II}, then cooled and treated with H_2O (10 ml) and with conc. H_3PO_4 (3 ml), and the Fe^{II} formed (corresponding to the amount of alkylnitrosohydrazine) was titrated with $KMnO_4$ soln. The coeff. of variation were <1·8% (17 to 20 results). M. P.

1C34. **Polarographic determination of acid impurities in dimethylformamide.** Mairanovskii, S. G.; Gul'tyai, V. P.; and Kaplunskii, G. D. (N. D. Zelinskii Inst. Org. Chem., Acad. Sci. USSR, Moscow). *Zavod. Lab.*, 1977, **43** (6), 655–656 (in Russian).—Mix 5 ml of the sample and 1 ml of a 10mM soln. of 4,4′-bipyridyl in pure dimethylformamide (**I**), dilute to 10 ml with M-$LiNO_3$ in **I**, de-gas the mixture, and record a polarogram from −1·4 to −2·2 V (*vs.* a silver electrode). Determine the ratio of the height of the first peak to the total height of all the peaks; this ratio is directly proportional to the acid concn., and the concn. of acid impurities is calculated from a calibration graph prepared with up to 0·6 mM-acetic acid in pure **I**. A. S. W.

1C35. **Retention indices of n-aliphatic sulphur compounds.** Moeckel, H. J.; and Zolg, Monika (Hahn-Meitner Institut fuer Kernforschung Berlin G.m.b.H., Bereich Strahlenchemie, Berlin, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **285** (1), 45–46 (in German).—The g.l.c. retention indices (corrected for dead time) for 27 disulphides, 12 sulphides, 8 thiols and 6 dithiols on SE-30 and Carbowax 20M are listed. J. K. C.

1C36. **Titration of thioethers.** Casalini, Claudio; Cesarano, Giulia; and Mascellani, Giuseppe (Res. Lab., Alfa Farmaceutici S.p.a., Bologna, Italy). *Anal. Chem.*, 1977, **49** (7), 1002–1004.—The sample is titrated potentiometrically, in the presence of (preferably) KBr in aq. 70% acetic acid, with Pb tetra-acetate soln. During titration, Br^- catalyse the conversion of the thioether into an intermediate

species that is readily hydrolysed to the sulphoxide; the reaction mechanism is discussed. The method can be applied to alkyl and aryl thioethers and to cephalosporins. G. P. C.

1C37. **Non-aqueous redox determination of xanthates and organotrithiocarbonates with copper(II) nitrate in acetonitrile.** Verma, Balbir Chand; and Kumar, Swatantar (Chem. Dept., Himachal Pradesh Univ., Simla, India). *Microchem. J.*, 1977, **22** (2), 149–156.—Titrations of xanthates or trithiocarbonates with 0·05N-$Cu(NO_3)_2$ in acetonitrile are made, either visually with *NN*-diphenylbenzidine as internal indicator (sharp change from pale yellow to blue for xanthates and from red to blue for trithiocarbonates) or potentiometrically with use of a polished platinum-wire indicator electrode and a slightly modified s.c.e. or antimony electrode as reference. Results by both procedures for the K salts of 10 xanthates and 7 trithiocarbonates show high accuracy for 10- and 40-mg samples. The reaction mechanism is discussed. W. J. B.

1C38. **Use of indicators in titration of mercaptoacetic acid with iodine.** Muralikrishna, U.; and Prasad, N. Satyanarayana (Dept. Chem., Andhra Univ., Waltair, India). *Acta Cienc. Indica*, 1976, **2** (2), 131–132.—Six thiazine dyes were used as reversible indicators in the titration of mercaptoacetic acid with 0·01N- and 0·1N-iodine. The endpoint colour change was from blue to green when methylene green (C.I. Basic Green 5), New methylene blue (C.I. Basic Blue 24), toluidine blue (C.I. Basic Blue 17) and Azure A or C [3-amino-7-(dimethylamino)- or 3-amino-7-(methylamino)-phenothiazin-5-ium chloride, respectively] were used, and was from violet to grey with thionine. The average error was 0·3 to 0·4%, and the blank correction was 0·06 ml of 0·01N-iodine for 0·3 ml of 0·01% indicator soln. CHEM. ABSTR.

1C39. **Determination of impurities in diethylzinc.** Solomatin, V. S.; Dzhupii, L. S.; and Nikolina, S. N. *Zavod. Lab.*, 1977, **43** (6), 685–686 (in Russian).—Cool the sample (1 ml) in liquid N, add 2 ml of ethanol, and when the reaction has subsided, add 3 ml of HNO_3 (1 : 1) dropwise, with cooling if necessary; evaporate the mixture under an i.r. lamp, and ignite the residue at 550° for 40 min. Mix 200 mg of the resulting oxide with 50 mg of graphite containing 10% of NaCl, and submit the mixture to spectrography in a d.c. arc. Use mixtures containing 8 and 40 p.p.m. of each impurity, prepared in a similar manner, as standards. Equations are given for calculating the concn. of each impurity, and measurement wavelengths and typical limits of detection are reported for Cu, Cd, Al, Fe, Ga, Sb, Bi, Cr, Mg and Mn. A. S. W.

1C40. **Gas-chromatographic determination of organic impurities in ethyl compounds of tin, tellurium and selenium.** Efremov, E. A.; Makarenko, V. G.; Filippov, E. P.; and Fedorov, V. A. *Zh. Anal. Khim.*, 1977, **32** (5), 1041–1043 (in Russian).—Impurities (≃0·01 to 2%) of ethyl ether and iodoethane in tetraethyltin, diethylselenium and diethyltellurium were determined by g.l.c. on a column (2 m × 4 mm) packed with 15% of Carbowax 20M (for tetraethyltin), or 15% of silicone gum SKTFV 803, supported on Chromaton N AW-DMCS (particle size 0·16 to 0·2 mm), with helium containing <0·8 p.p.m. of H_2O as carrier gas and thermal-conductivity detection. M. P.

1C41. **High-pressure liquid chromatographic separations of some transition metal cluster complexes.** Enos, Clay T.; Geoffroy, Gregory L.; and Risby, Terence H. (Dept. Chem., Pennsylvania State Univ., University Park, Pa. 16802, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (2), 83–84.—The compounds, consisting of Ru, Ir and Co carbonyl complexes, are separated on columns (100 mm × 2·1 mm) of Corasil II C_{18} with aq. 75 to 80% methanol as mobile phase at flow rates from 0·9 to 5·2 ml min^{-1}. An u.v. detector set at 254 nm is used. The method was applied to the analysis of products of the photolysis of an Fe - Co - CO - triphenylphosphine complex. M. J. SA.

1C42. **Plasma chromatography of isomeric dihalogenated benzenes.** Carr, Timothy W. (International Business Machines Corp., Poughkeepsie, N.Y. 12602, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (2), 85–88.—The BETA VII plasma chromatograph (*cf.* Cohen and Karasek, *Anal. Abstr.*, 1971, **20**, 3504) was used in this study. In this technique ion - molecule complexes in a N carrier gas are formed through a series of ion - neutral molecule reactions initiated by 60-keV β-particles from a ^{63}Ni foil. The procedure was applied to the *o*-, *m*- and *p*-isomers of the difluoro-, dichloro-, dibromo- and di-iodo-benzenes, and to some nitrobenzenes. Positive-mode spectra illustrated the effects of isomeric substitution on the drift-time of ions. In the negative-mode spectra, only the halogen ions were detected. M. J. SA.

1C43. **Study of retention behaviour of alkylphenols in normal and reversed-phase liquid chromatography and application to analysis of complex phenolic mixtures in conjunction with gas chromatography.** Callmer, K ; Edholm, L.-E.; and Smith, B. E. F. (Dept. Tech. Anal. Chem., Chem. Centre, Lund, Sweden). *J. Chromatogr.*, 1977, **136** (1), 45–62.—The liquid chromatography of 33 alkylphenols was studied in four systems, *viz*, 1,2,3-tris(cyanoethoxy)propane and Fractonitril VI (Merck) liquid phases on Porasil C (37 to 74 μm), and Cyano Sil-X-I (Perkin-Elmer) and μBondapak C_{18} (Waters Assoc.) bonded-phase packings. In the first two systems 2,2,4-trimethylpentane was used as mobile phase, and in the other two systems 0·5% isopropyl alcohol in 2,2,4-trimethylpentane and 60% aq. ethanol, respectively, were used. In the first three systems the elution order was di-, mono- and non-*ortho*-substituted phenols, while on μBondapak C_{18} the number of substituted alkyl C atoms determined the retention. A combination of normal and reversed-phase liquid chromatography and capillary-column g.l.c. aids the separation and identification of complex mixtures of alkylphenols. M. J. SL.

1C44. **Separation of diastereoisomers of the vinylic oligomers 2,4-dibenzoylpentane, 1,3,5-tribenzoylhexane and 2,4,6-tribenzoylheptane by high-resolution gel-permeation chromatography with recycling.** Lesec, J.; Salvin, R.; Balard, H.; and Meybeck, J. (Lab. Physicochim. Macromol., Ecole Super. Phys. et Chim., Paris, France). *Analusis*, 1977, **5** (5), 220–222 (in French).—These separations were studied with use of three columns, each 3 ft. × 0·375 in. i.d., in series and each packed with 100-Å μStyragel (particle size 10 μm), with isopropyl ether (2 ml min^{-1}) as solvent. For 100 or 600 mg of sample 4 or 8 passes, respectively, were necessary to achieve complete separation. B. D. H.

1C45. **Sensitivity of an electron-capture detector towards aromatic amides of polyfluorocarboxylic acids.** Pashkevich, K. I.; Kirichenko, V. E.; Postovskii, I. Ya.; Kulikova, G. S.; Bil'dinov, K. N.; and Deev, L. E. (Inst. Chem. Urals Sci. Centre, Acad. Sci. USSR, Sverdlovsk). *Zh. Vses. Khim. O-va.*, 1977, **22** (2), 225–226 (in Russian).—Sixteen such amides (including a trifluoroacetanilide and 4′-bromo- and -chloro-pentafluorobenzanilides) were examined by g.l.c. with use of an electron-capture detector (operated at 250°) and a U-shaped glass column (1 m × 3 mm) filled with 5% of XE-60 on Chromaton N-AW (75 to 90 mesh) and operated at 200°, with N as carrier gas (45 ml min^{-1}). Threshold sensitivity levels are given for the 16 compounds examined. M. R. M.

1C46. **[Gas-chromatographic] identification of isomeric nitro-derivatives of *p*-chlorobenzotrifluoride [4-chloro-ααα-trifluorotoluene] by using retention data.** Bratolyubova, A. G.; Korol', A. N.; and Lozinskii, M. O. (Inst. Org. Chem., Acad. Sci. Ukr. SSR, Kiev). *Zh. Anal. Khim.*, 1977, **32** (6), 1251–1254 (in Russian).—Molar entropy and molar enthalpy of solution of nine such compounds were calculated from g.c. retention data obtained by use of columns packed with (*i*) a non-polar stationary phase (SE-30) and (*ii*) a polar phase [poly(ethanediol adipate)]. The data were used to identify 2-chloro-3-nitro- and 5-chloro-2,3-dinitro-ααα-trifluorotoluene as impurities in 4-chloro-3-nitro-ααα-trifluorotoluene, which is used in production of the herbicide nitrofor (4-diethylamino-ααα-trifluoro-3,5-dinitrotoluene). M. P.

1C47. **Polarographic determination of diphenyl disulphide and diethyl disulphide.** Reishakhrit, L. S.; Argova, T. B.; Zhustareva, S. S.; and Krunchak, V. G. *Vestn. Leningr. Univ.*, 1977, (4), *Fiz., Khim.*, (1), 149–151 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (17), Abstr. No. 17G292.

1C48. **Spectrophotometric assay of chloramine T by using sulphanilamide.** Trieff, Norman M.; Ramanujam, V. M. Sadagopa; and Forti, G. Cantelli (Dept. Preventive Med. and Community Health, Univ. Texas Med. Branch, Galveston, Tex. 77550, U.S.A.). *Microchem. J.*, 1977, **22** (2), 222–225.—The absorbance of the reddish-brown complex formed in the reaction between chloramine T and sulphanilamide (in large excess) at pH 7·4 (phosphate buffer soln.) and 25° to 50° is measured at 530 nm. The calibration graph is rectilinear for 40 to 500 μg ml^{-1} of chloramine T, and the method is ≃10 times as sensitive as more conventional methods. W. J. B.

1C49. **Polarographic reduction of mixed-ligand complexes of zinc and cadmium with ethylxanthate and 2,2′-bipyridyl.** Budnikov, G. K.; Ulakhovich, N. A.; and Sharapova, T. S. (Kazan State Univ., USSR). *Zh. Obshch. Khim.*, 1977, **47** (6), 1219–1221 (in Russian).—These compounds, which have the composition $M(C_2H_5OCS_2)_2$.Bipy (where Bipy = 2,2′-bipyridyl), give three polarographic waves, the first two corresponding to formal discharging of the metal atom and the third (in the more negative region) to reduction of the 2,2′-bipyridyl. The oscillopolarograms each show three peaks, corresponding to the three waves. With mM soln. of the compounds and 0·05M-tetraethylammonium perchlorate as supporting electrolyte, the $E_{\frac{1}{2}}$ values for the Zn complex are −0·78, −0·94 and −1·35 V; those for the Cd complex are −0·56, −0·81 and −1·55 V. V. S.

1C50. **Sub-part-per-trillion [10^{12}] detection of polycyclic aromatic hydrocarbons by laser-induced molecular fluorescence.** Richardson, J. H.; and Ando, M. E. (Gen. Chem. Div., Lawrence Livermore Lab., Univ. California, Livermore, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 955–959.—Naphthalene, anthracene, fluoranthene and pyrene (at levels of 1·3, 4·4, 1·0 and 0·5 pg g^{-1}, respectively), and benzene (19 pg g^{-1}), were detected in aq. soln. by means of a system incorporating a N- and dye-laser source, a monochromator, a boxcar integrator and an *X* - *Y* recorder. Excitation and emission wavelengths ranged from 254 to 287 nm and from 302 to 450 nm, respectively. The fluorescence intensity was rectilinearly related to concn. (in some instances, over 6 orders of magnitude). G. P. C.

1C51. **Gas-chromatographic analysis of pyridine bases.** Sednevets, L. G.; and Pankov, A. G. (Sci.-Res. Inst. Monomers for Synth. Rubber, Yaroslavl', USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 1039–1041 (in Russian).—Retention times (relative to pyridine) are tabulated for 76 alkyl- and alkenyl-pyridines. The values were obtained by g.l.c. at 120° on a column (3 m × 3 mm) packed with 10% of polyoxyethylene glycol 1000 on Chromaton (pre-treated with ethanolic 2% NaOH), with a carrier-gas flow rate of 20 ml min^{-1} and flame ionisation detection. M. P.

1C52. **Ion-pair partition systems for chromatographic analysis of hydrophilic organic bases.** Soczewinski, Edward; Ratajewicz, Danuta; and Rojowska, Maria (Dept. Inorg. and Anal. Chem., Inst. Chem. Sci., Med. Acad., Lublin, Poland). *Acta Pol. Pharm.*, 1977, **34** (2), 181–185.—Several 4-acyl-4-phenyl-1-(3-piperidinopropyl)piperidines were separated on Whatman No. 4 paper by descending development with solvents of the type cyclohexane - $CHCl_3$ - buffer soln. or $CHCl_3$ - bis-(2-ethylhexyl) hydrogen phosphate - 0·1M-HCl; the spots were located with Dragendorff reagent. R_M values indicated that solvents of the second type cited are more efficient and selective, and are suitable for the chromatographic separation of hydrophilic organic bases (*e.g.*, various pharmaceutical compounds and their metabolites) of analogous molecular structure to the named piperidines. F. R. B.

1C53. **Chemistry of carbazole derivatives. CXII. Determination of carbazole and anthracene in the presence of each other.** Zherebtsov, I. P.; Lopatinskii, V. P.; Yudina, S. P.; Pirogov, V. D.; and Zorina, T. N. *Izv. Tomsk. Politekh. Inst.*, 1976, (274), 97–99 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G297.

1C54. **Preparation of high-purity organic compounds. XI. Determination of trace anthracene and phenanthrene in carbazole by fluorescence lifetimes and time-resolved fluorescence spectra with solid materials.** Iwashima, Satoshi; Sawada, Tadanobu; and Aoki, Junji (Dept. Chem., Fac. Sci. and Technol., Meisei Univ., Hodokubo, Hino-shi, Tokyo, Japan). *Bunseki Kagaku*, 1977, **26** (6), 401–406 (in Japanese).—Evaporated thin films from carbazole containing various amounts of pure anthracene or phenanthrene were prepared and the limits of determination were investigated. The concn. of anthracene could be measured down to 0·1 μmol mol^{-1} from the fluorescence spectra and to 0·01 μmol mol^{-1} from the time-resolved fluorescence spectra. The fluorescence lifetime of anthracene was too close to that of

carbazole be used for the determination. The concn. of phenanthrene in carbazole could be measured down to $<0{\cdot}1$ mmol mol^{-1} from the fluorescence spectra, to $0{\cdot}1$ μmol mol^{-1} from the fluorescence lifetimes and to 1 μmol mol^{-1} by the time-resolved fluorescence spectra. (*Cf. Anal. Abstr.*, 1976, **31**, 2C59.)

1C55. **Mass spectra of macroheterocyclic chelates and their analytical use.** Kuzanyan, R. S.; Poponova, R. V.; Chupakhin, M. S.; Dziomko, V. M.; Tomsons, U. A.; and Fedosyuk, L. G. (All-Union Sci.-Res. Inst. Reagents and High-purity Chemicals, Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 884–891 (in Russian).—It is shown that chelates involving 14-membered rings containing 6 atoms of N, with Ni^{II} or Cu^{II} as the central atom, give simple mass spectra that can be used for identifying the chelates and checking their purity during preparation from, *e.g.*, bisazo-compounds involving condensed 3-aminopyrazole and benzene rings. With an AEI MS-702 mass spectrometer, the lower limit of determination of an individual chelate in a mixture was 10 p.p.m. M. P.

Industrial products

Including petroleum and its products, fuels, detergents, essential oils, cosmetics, dyes, fibres, plastics, resins, paints, elastomers, leather, photographic materials and explosives

1C56. **Polarographic determination of carbonyl sulphide and carbon disulphide as impurities in natural gas.** Pekhota, F. N.; Kamenev, A. I.; and Byr'ko, V. M. (All-Union Sci.-Res. Inst. Natural Gas, Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1210–1213 (in Russian).—The impurities were concentrated and converted into the corresponding thiocarbamic acid derivatives by passing the sample gas (at $\simeq 5$ l h^{-1}) through ethanolic 5% diethylamine (20 ml) at 0°. The resulting soln. was made 0·2M in $LiNO_3$ and subjected to polarography from $-0{\cdot}9$ to $+0{\cdot}2$ V *vs.* the silver - AgCl electrode. Two waves, corresponding to oxidation of the reaction products of COS and CS_2 with diethylamine and having respective $E_{\frac{1}{2}}$ values of $-0{\cdot}26$ V and $-0{\cdot}47$ V, were observed. The heights of the waves were rectilinearly related to concn. of COS and CS_2 in the ranges 1 to 10 μg l^{-1} and 10 to 120 μg l^{-1}, respectively; the molar ratio of CS_2 to COS should not exceed 4. Hydrogen sulphide and thiols interfere, and should first be removed by bubbling the sample through 30% $CdCl_2$ soln. in 0·1M-HCl and then through aq. 3% $AgNO_3$. The limits of determination were 0·1 μg l^{-1} for COS and 0·5 μg l^{-1} for CS_2. The coeff. of variation were $<7\%$ (10 to 15 results) for 50 mg m^{-3} of COS and 1 mg m^{-3} of CS_2 in natural gas. M. P.

1C57. **Determination of nitrogen in petroleum fractions by combustion with chemiluminescence detection of nitric oxide.** Drushel, Harry V. (Res. and Dev. Lab., Exxon Co., Baton Rouge, La. 70821, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 932–939.—The detector described by Parks (27th Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Cleveland, Ohio, March 1976) has been applied to the analysis of some naphtha fractions containing a few p.p.m. of N and to residual fuels or asphaltenes containing as much as 1% or more of N. The results agreed to within $\pm 5\%$ with those by the Kjeldahl method. The short-term coeff. of variation (2σ criterion) was $\simeq 3\%$, while the long-term value was 6%. G. P. C.

1C58. **Study of oxygen compounds in middle [petroleum] fractions.** Polyakova, A. A.; Chertkov, Ya. B.; Zimina, K. I.; Vykhristyuk, N. I.; Nekhamkina, L. G.; Kogan, L. O.; Vasilenko, G. V.; Kirsanova, T. I.; and Semanyuk, R. N. *Tr. Vses. Nauchno-Issled. Inst. Pererab. Nefti*, 1976, (19), 3–17 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (17), Abstr. No. 17G249.

1C59. **Determination of chlorides in petroleum.** Lobov, B. I.; Vinogradova, I. G.; Krupnik, V. B.; Kabardin, G. A.; Nigmatullin, R. M.; and Molochnyi, V. B. (Gertsen State Univ., Leningrad, USSR). *Zavod. Lab.*, 1977, **43** (6), 658–659 (in Russian).—Extract the salts into H_2O, and make the extract 0·01M in H_2SO_4. Determine Cl^- in this soln. by measuring the e.m.f. (silver - AgCl indicating electrode; glass reference). The calibration graph (e.m.f. *vs.* —ln concn.) is rectilinear down to 6 μg-ion l^{-1} of Cl^-. A. S. W.

1C60. **Determination of traces of lead in gasoline by an inverse voltammetric method.** Sebestyen, B. *Dunai Koolaj*, 1976, (1), 67–77 (in Hungarian).—The sample is digested to convert organolead compounds into Pb^{2+}, which are extracted into HCl and then subjected to voltammetry at $-0{\cdot}42$ V in 0·1M-HCl medium, with use of a hanging-mercury-drop indicator electrode and a s.c.e. as reference. Results are obtained by reference to a calibration graph based on samples of gasoline containing known amounts of Pb or, preferably, by a standard-addition method. The relative error is $\pm 6\%$, and the limit of detection is 1 p.p.b. of Pb. HUNG. TECH. ABSTR.

1C61. **Determination of total sulphur in naphthas.** Fernandez, T.; Rocha, J. M.; Rufino, N.; and Garcia Luis, A. (Compania Espanola de Petroleos SA, Santa Cruz de Tenerife, Canary Isl.). *Analyst (London)*, 1977, **102** (1215), 414–423.—In this method the sample is vaporised in CO_2 and burnt in O, combustion products are absorbed in H_2O_2 soln. and the SO_4^{2-} formed are titrated, in aq. ethanol, with 0·01M-$Ba(ClO_4)_2$ with use of a h.f. conductometric titrator. Attention is drawn to the volatility of H_2SO_4, which can cause losses at the evaporation stage. The method has been applied to samples containing 0·4 to 50 μg of S. The apparatus and circuitry used are shown in detail. R. W.

1C62. **Method for selected polyaromatics in mineral oil by linear elution chromatography as a rapid alternate for Food and Drug Administration method 121.2589 (c) (3).** Skinner, G. R.; and Smith, R. E. (Kerr-McGee Refining Corp., Oklahoma City, Okla. 73125, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (2), 75–78.—By means of special glass equipment (illustrated), the polycyclic hydrocarbons are isolated on a 1% de-activated alumina column. Lower aromatic hydrocarbons are separated from tetracyclic and higher aromatic hydrocarbons by chromatography on a 1% de-activated alumina column and on a MgO - Celite column, with a system of eluents of increasing polarity. Components are detected (in methanol - ethyl ether soln.) at various wavelengths. This method is intended for testing mineral oil for use in applications where incidental contact with food may occur. M. J. SA.

1C63. **Investigation of the compounds causing the offensive smell of paraffin wax.** Siklos, Pal; and Nagy, Lajos (Dept. Chem. Technol., Tech. Univ., Budapest, Hungary). *Period. Polytech. Chem. Eng.*, 1977, **21** (1), 73–78.—The wax sample

(40 to 80 g) was heated, with frequent shaking, with 20 to 25% (w/w) of molecular sieve (Molfilit KX-130 or Linde 13X) at 80° to 100° for 3 h. The molecular sieve was separated by decantation, washed with heptane and then extracted with acetone (250 ml) for 6 h. The malodorous yellow solid remaining after evaporation of the acetone was examined by i.r. and n.m.r. spectrometry. The presence of compounds containing carbonyl and hydroxy-groups and of olefinic and aromatic hydrocarbons was indicated. L. B.

1C64. **Determination of vanadium in Athabasca bitumen and other heavy hydrocarbons by visible spectrometry.** Funk, Edward W.; and Gomez, Ernesto (Corporate Res. Lab., Exxon Research and Engineering Co., Linden, N.J. 07036, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 972–974.—The method was based on that described by Patel (*Anal. Abstr.*, 1974, **27**, 3375) and was extended to the analysis of tar-sand bitumen that had been de-asphalted under various conditions; a separate correlation had to be developed for each hydrocarbon. Graphs of V concn. *vs.* absorbance are shown for various samples. G. P. C.

1C65. **Separation and determination of polynuclear aromatic hydrocarbons by high-performance liquid chromatography. I.** British Carbonization Research Association (Wingerworth, Chesterfield, Derbys., England). *Carbonization Res. Rep.*, No. 28, 1976. Pp. 20.—Such hydrocarbons having four to seven rings are separated by h.p.l.c. at 1200 p.s.i. on a column (30 cm × 4·5 mm) of Partisil (5 μm), with hexane as mobile phase and use of u.v. detection (variable wavelength); the preparation of the column is described. The method was used, in conjunction with t.l.c. on Bentone 34 - Kieselgel G, for identifying 25 compounds in the cyclohexane-soluble fraction of pitch; its use for the specific determination of benzo[*a*]pyrene, with monitoring at 385 nm, is also described. B. H.

1C66. **BCRA review.** British Carbonization Research Association (Carbonization Res. Centre, Wingerworth, Chesterfield, Derbys., England). *BCRA Rev.*, 1976, **3** (1), 1–107.—The section of this publication dealing with gas and by-products (pp. 67 to 84) includes 3 references to methods for analysing hydrocarbons, 1 to the determination of S in organic substances, 1 to analysis to coke-oven gas, 10 to that of light oils, 9 to that of middle oils, 2 to that of pitch, 11 to that of tar acids and 7 to that of tar bases. The section on pollution (pp. 95 to 104) contains 16 references of analytical interest. B. H.

1C67. **BCRA review.** British Carbonization Research Association (Carbonization Res. Centre, Wingerworth, Chesterfield, Derbys., England). *BCRA Rev.*, 1976, **3** (2), 109–193.—The sections of this publication dealing with coal (pp. 109 to 111), coke (p. 117) and pollution (pp. 185 to 191) contain, respectively, 4, 1 and 9 references of analytical interest. The section on gas and by-products contains 2 references to analysis of gas, 4 to that of light oils, 9 to that of middle oils, 2 to that of pitch, 4 to that of tar acids and 5 to that of tar bases. B. H.

1C68. **BCRA review.** British Carbonization Research Association (Carbonization Res. Centre, Wingerworth, Chesterfield, Derbys., England). *BCRA Rev.*, 1976, **3** (3), 195–283.—The sections of this publication dealing with coal (pp. 195 to 197), coke (pp. 204 to 206) and pollution (pp. 274 to 282) contain, respectively, 6, 1 and 10 references of analytical interest. The section on gas and by-products (pp. 250 to 262) includes 1 reference to general analysis, 1 to analytical methods for tar, 7 to analysis of light oils, 11 to that of middle oils, 5 to that of tar acids and 2 to that of tar bases. B. H.

1C69. **BCRA review.** British Carbonization Research Association (Carbonization Res. Centre, Wingerworth, Chesterfield, Derbys., England). *BCRA Rev.*, 1976, **3** (4), 285–378.—The sections of this publication dealing with coal (pp. 285 to 289) and pollution (pp. 369 to 376) contain, respectively, 6 and 11 references of analytical interest. The section on gas and by-products (pp. 347 to 358) includes 5 references to general analytical methods, 6 to analysis of light oils, 8 to that of middle oils, 8 to that of tar acids and 5 to that of tar bases. B. H.

1C70. **BCRA review.** British Carbonization Research Association (Carbonization Res. Centre, Wingerworth, Chesterfield, Derbys., England). *BCRA Rev.*, 1977, **4** (1), 1–81.—The sections of this publication dealing with coal (pp. 1 to 3) and pollution (pp. 69 to 79) contain, respectively, 6 and 17 references of analytical interest. The section on gas and by-products (pp. 47 to 57) contains 3 references to the analysis of tar, 7 to that of light oils, 10 to that of middle oils, 2 to that of pitch, 5 to that of tar acids and 2 to that of tar bases. B. H.

1C71. **Indirect determination of carbon disulphide in coke-oven gas by atomic-absorption spectrophotometry.** Coutinho, Carlos A.; De Sousa, Jose G.; and Saito, Paulo Y. *Metal. ABM*, 1976, **32** (227), 683–685 (in Portuguese).—The CS_2 was absorbed in alcoholic KOH (to form K ethylxanthate) after the H_2S had been removed by pptn. with $CdSO_4$ soln. The resulting soln. was treated with a Cu salt to form Cu ethylxanthate, which was pptd., and the amount of Cu present was determined by a.a.s. after decomposition of the ppt. The sensitivity of the method was 9 mg m^{-3} of CS_2. CHEM. ABSTR.

1C72. **Chromatographic analysis of non-ionic surface-active agents of the oxyethylated fatty alcohol type.** Kofanov, V. I.; and Klimenko, N. A. (Inst. Colloid and Water Chem., Acad. Sci. Ukr. SSR, Kiev). *Zavod. Lab.*, 1977, **43** (6), 668–670 (in Russian).—Commercial samples (mixtures of ethers) are fractionated on a preparative column, and the fractions are further analysed by t.l.c. *Procedure*—Apply the sample, with toluene, to a column (130 cm × 2·8 cm) of silica gel (particle size 0·05 to 0·2 mm), and elute with acetone - toluene - H_2O (the proportions vary according to the number of oxyethylene-groups) at 1 ml min^{-1}, collecting 10-ml fractions. Submit each fraction (0·05 ml) to t.l.c. on a layer of silica gel - gypsum (14 : 1) with the appropriate acetone - toluene - H_2O mixture as mobile phase, and, after drying the chromatogram in warm air, locate the spots by spraying with a Dragendorff reagent. The effect of the type of sorbent on the R_F values is indicated. A. S. W.

1C73. **Field-desorption and electron-impact mass spectra of ionic dyes.** McEwen, C. N.; Layton, S. F.; and Taylor, S. K. (Central Res. and Dev. Dept., E. I. du Pont de Nemours and Co., Wilmington, Del. 19898, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 922–926.—As an example, the dyes were extracted

from photographic film with methanol (after removal of the emulsion), and the extract was concentrated and purified on a column of silica gel at pH 7 by successive elution with benzene, $CHCl_3$ and $CHCl_3$ - methanol (4 : 1). Each eluate fraction was submitted to m.s. (in some instances after a further purification by paper chromatography and/or h.p.l.c.). The field-desorption technique gave good results for the identification of sub-mg amounts of dyes, but was adversely affected by the presence of impurities. Relative ion intensities and *m/e* values are tabulated for various dyes, and the respective merits of the two techniques are discussed. Application to the identification of ionic dyes in polluted water or living tissue is indicated. G. P. C.

1C74. **Triphenylmethane dyes as reagents for the extraction - spectrophotometric determination of various anions. XX. Determination of iodides of basic dyes with simultaneous oxidation by iodate and chloramine B.** Ramanauskas, E.; Zalieckiene, E.; Bunikiene, L.; and Markeliunaite, J. *Liet. TSR Aukst. Mokyklu Mokslo Darb., Chem. Chem. Technol.*, 1976, (18), 41–45 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (16), Abstr. No. 16G168.

1C75. **Use of thin-layer chromatography in studying the nitration of 9-oxofluorene-4-carboxylic acid.** Migachev, G. I.; Grekhova, N. G.; Lyande, Yu. V.; and Dubina, E. M. (Moscow Sci.-Res. Inst. Org. Intermediates and Dyes, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1254–1256 (in Russian).—Fluoren-9-one, 9-oxofluorene-4-carboxylic acid (**I**), the 5-nitro-, 7-nitro-, 2,5-dinitro-, 2,7-dinitro- and 2,5,7-trinitro-derivatives of **I** and 2,4,5,7-tetranitrofluoren-9-one can be separated on silica gel by using benzene - acetone (4 : 1) as the mobile phase. The sample (20 to 40 μg of a compound) is applied as a soln. in acetone (2 to 3 μl). The zones are detected as yellow to brown spots by spraying the air-dried chromatogram with 1% $SnCl_2$ soln. in 5% HCl, drying at ambient temp., and spraying with 0·5% 4-dimethylaminobenzaldehyde soln. in ethanol - M-HCl (1 : 1). Estimation of the compounds by visual comparison of the colour intensities with those for standards is possible; the lower limit is 2 μg. The method has been used to study the products of nitration under various conditions. M. P.

1C76. **Determination of resin content of wood from softwoods.** Nelson, P. J.; Murphy, P. I.; and James, F. C. (Forest Products Lab., Div. Chem. Technol., CSIRO, South Melbourne, Australia). *Appita*, 1977, **30** (6), 503–504.—The wood sample is split into matchstick-sized pieces and freeze-dried at low pressure (< 0·07 kPa) for 3 days. After milling to pass a 0·5-mm screen, the wood (45 g) is heated for 16 h with light petroleum (400 ml) in a Soxhlet apparatus. The wood is then dried at 105° for 16 h and weighed. The resin soln. is filtered and evaporated to dryness under reduced pressure. The resin residue is dissolved in light petroleum and the soln. is evaporated; then the flask is heated (50°) at low pressure (0·11 kPa) to constant wt. and the resin content is calculated. A. B. K.-S.

1C77. **Gas-chromatographic determination of the products of interaction of laevoglucan with propene oxide.** Brangulis, K.; Pernikis, R.; Lazdina, B.; and Surna, J. (Inst. Wood Chem., AN Latv. SSR). *Latv. PSR Zinat. Akad. Vestis, Kim. Ser.*, 1976, (3), 276–278 (in Russian).—The products were analysed on a column (0·8 to 1·2 m × 6 mm o.d.) packed with 15% of Apiezon L and 5% of XE-60 on Chromaton NAW-DMCS (0·100 to 0·125 mm) and temp.-programmed from 180° to 260° at 8° min^{-1}, or from 240° to 300° at 4° min^{-1}, with He as carrier gas (50 ml min^{-1}) and a plasma ionisation detector; trimethylaevoglucan was used as internal standard. V. S.

1C78. **Use of a support in radio-isotopic X-ray fluorescence analysis of paper.** Lisnyanskii, Kh. A. (Central Res. and Design Inst. for Cellulose - Paper Ind., Leningrad, USSR). *Zavod. Lab.*, 1977, **43** (6), 680–682 (in Russian).—The procedure described for determining TiO_2 used as filler is claimed to reduce the error due to variation in the weight of the paper. An incident beam from a monochromatic 50-mCi ^{55}Fe source falls on the upper surface of the sample (in the form of a flat sheet), which is supported on a flat base prepared from the same fibre as the sample and containing a similar amount of filler (the combined thickness of the sample and base is greater than the saturation thickness). The excited secondary Ti radiation is counted with a Xe - CH_4 proportional counter. An expression is derived for calculating the TiO_2 content. A. S. W.

1C79. **Potentiometric method for determining reducing capacity of sulphite liquor components.** Bogolitsyn, K. G.; Bokhovkin, I. M.; Prokshin, G. F.; Krunchak, V. G.; and Rodichev, A. G. *Izv. Vyssh. Uchebn. Zaved., Lesn. Zh.*, 1976, **19** (4), 109–113 (in Russian).—A potentiometric method for determining reducing capacities of pulping liquor components, based on changes in the redox potentials of reversible systems [*e.g.*, $K_4Fe(CN)_6$ - $K_3Fe(CN)_6$, Fe^{II} - Fe^{III} salts and Ce^{III} - Ce^{IV} salts], has been used for studying spent sulphite liquor. The sensitivity of the method with $K_4Fe(CN)_6$ - $K_3Fe(CN)_6$ depended on the ratio of oxidised and reduced forms, their concn. and pH. Different systems could be used for selectively determining components in the liquor, *e.g.*, Ce^{III} - Ce^{IV} for determining lignin. The $K_4Fe(CN)_6$ - $K_3Fe(CN)_6$ system could be used in strongly alkaline soln. for determining total SO_2 in sulphite liquors. Abstr. Bull. Inst. Pap. Chem.

1C80. **Determination of COD, BOD and TOC [total organic carbon] in the filtrate from suspensions of pulp, paper and waste paper.** Gottsching, L.; Dalpke, H. L.; and von der Heyde, H. *Papier (Darmstadt)*, 1977, **31** (1), 1–11 (in German).—The extension of the present analysis of effluents for salt content, COD, BOD and TOC to include determination of the products going into paper manufacture, *i.e.*, water, fibre and chemical auxiliaries, is discussed. Sample preparation of pulp, paper or waste paper for determining COD, BOD and TOC in suspension filtrates is outlined. Some COD values are presented for various mechanical and chemical pulps, groundwood-free and groundwood-containing papers, and waste paper. Abstr. Bull. Inst. P. p. Chem.

1C81. **New procedures in quantitative fibre analysis.** Schiller, Wolfgang; and Zimmer, Hubertus (Bundesanst. Materialpruefung, Berlin, W. Germany). *Melliand Textilber.*, 1977, **38** (4), 307–309 (in German).—Procedures for the analysis of mixtures of fibres by selective extraction of the components are considered. For removing polyester fibres, hot extraction with hexafluoropropan-2-ol is proposed, and for removing poly(vinyl chloride) fibres, hot extraction with CS_2 - acetone (111 : 89) or dioxan. In all instances, correction needs to be made to allow for

the solubility of the other fibre components (correction factors are tabulated). Steel fibres can also be determined, in some instances, by dissolution of the organic fibres in hexafluoropropan-2-ol.
L. A. O'N.

1C82. **Analysis of textile oils and other processing additives.** Fletcher, J. C. (Wira, Headingley Lane, Leeds, England). *Text. Manuf.*, 1977, (2), 36–38.—Methods available for such analyses are reviewed. Initial separation can be on the basis of polarity by partitioning the extract between, *e.g.*, hexane and 50% aq. ethanol, followed by t.l.c. on silica gel for the further separation of, *e.g.*, fatty components and ethoxylated materials, or by column chromatography on alumina or silicic acid. The fractions can be identified by i.r. spectrophotometry or by derivative formation and g.c. I. R. H. C.

1C83. **Polarographic determination of titanium dioxide in man-made fibre products and raw materials.** Gega, Henryk; Moniuk, Danuta; and Wojcik, Zofia (Text. Ind. Central Lab. South, Bielsko-Biala, Poland). *Przegl. Wlok.*, 1977, **31** (3), 143–144, 164 (in Polish).—Fuse the residue remaining after combustion of the sample (2 g) with $KHSO_4$ (2 g) in air, dissolve the cooled melt in 50% H_2SO_4 (10 ml) with heating if necessary, and dilute the soln. to 30 ml. Mix a 5-ml portion of this soln. with 0·2M-oxalic acid (15 ml), and remove O before recording the TiO^{2+} reduction wave between 0 and −2 V *vs.* the s.c.e.; obtain the result from a calibration graph. The standard deviation for a sample of polyester containing 0·6% of TiO_2 is ±0·0066% (10 determinations) and compares favourably with values for gravimetric (±0·0119%) and spectrophotometric (±0·0092%) determinations. P. B.

1C84. **High-performance size-exclusion chromatography of small molecules with columns of porous silica microspheres.** Kirkland, J. J.; and Antle, P. E. (E.I. du Pont de Nemours and Co., Central Res. and Dev. Dept., Wilmington, Del. 19898, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (5), 137–147.—Porous silica microspheres (6 to 11 μm diam. and with pore sizes ranging from 450 to 750 nm) were de-activated by exhaustive treatment with chlorotrimethylsilane. They were then packed into columns and used for separations of monomers, oligomers and low-mol.-wt. (<20,000) polymers. Efficiencies of up to 30,300 plates m^{-1} were measured with tetrahydrofuran as the mobile phase and toluene (total permeation) as solute. A wide range of organic and aq. solvents could be used, and separations were generally achieved within 20 min. M. J. Sʟ.

1C85. **Quantitative phosphorescence spectroscopy of polystyrene during photo-degradation and the significance of in-chain peroxides.** George, G. A.; and Hodgeman, D. K. C. (Materials Res. Lab., Maribyrnong, Vic., Australia). *Eur. Polym. J.*, 1977, **13** (1), 63–71.—Benzoyl end-groups in polystyrene were determined by phosphorescence spectroscopy at 77 K, emission being gathered at an angle of 45° from the excitation beam; 3-phenylpropiophenone (**I**) was used as standard. The calibration graph [phosphorescence intensity at 398 nm (corrected for thickness) *vs.* **I** content of the standard] was rectilinear. The change in mol. wt. of polystyrene during photo-oxidation was used to calculate the number of chain scissions per molecule; the number of carbonyl groups produced per scission was 1·31, or 0·65 for each new end-group, which was in reasonable agreement with the value expected from alkoxy-fragmentation after hydroperoxide thermolysis or photolysis. Commercial polystyrene was found to contain 0·14 carbonyl group per number-average chain before photo-oxidation; a study of the emission properties of polystyrene polymerised under O, and of hydroperoxidised polystyrene both before and after photolysis, suggested that the carbonyl groups in commercial polystyrene were formed from in-chain peroxides and not from hydroperoxides. I. R. H. C.

1C86. **Determination of labile chlorine in poly(vinyl chloride) with the aid of phenolysis reaction.** Robila, Gabriela; Buruiana, E. C.; and Caraculacu, A. A. (Inst. Macromol. Chem. 'Petru Poni', Iasi, Romania). *Eur. Polym. J.*, 1977, **13** (1), 21–24.—Tertiary and allylic chlorine, which comprises the labile chlorine in poly(vinyl chloride) (PVC), has been determined. The polymer (0·5 g) was heated with phenol (115 g) at 60° for 96 h. The excess of phenol was then decanted off and the polymer was washed with methanol and reprecipitated from cyclohexane - methanol (twice), dichloroethane - methanol (twice) and tetrahydrofuran - methanol. This purification procedure removed all unreacted phenol. The absorbance of a 1% soln. of polymer in tetrahydrofuran was measured at 285 nm and the calibration graph used was based on the absorbance of 3-(*p*-hydroxyphenyl)pent-1-ene. The present method has identified labile chlorine values (Cl_L) in PVC of between 0·04 and 0·19 Cl_L per 100 monomer units. An inverse relationship between Cl_L and mol. wt. has been found, suggesting a prevalence of end-allylic chlorine structures. I. R. H. C.

1C87. **Rapid analysis of starch graft copolymers.** Dennenberg, R. J.; and Abbott, T. P. (N. Reg. Res. Center, A.R.S., U.S.D.A., Peoria, Ill. 61604, U.S.A.). *J. Polym. Sci., Polym. Lett. Ed.*, 1976, **14** (1), 693–696.—Starch - vinyl graft copolymers, prepared from pearl corn starch with use of $(NH_4)_2Ce(NO_3)_6$ in N-HNO_3 as initiator, were analysed by oxidation with $HClO_4$ in anhyd. acetic acid. Quantitative decomposition and recovery of vinyl side-chain material were achieved by dissolving 2 g of the copolymer containing H_2O-insoluble side-chains in 100 ml of anhyd. acetic acid at 90° to 100° during 1 h; 2 ml of 60% $HClO_4$ was added, and, after completion of the reaction in 2 min, the reaction mixture was poured into ice - H_2O, and the precipitated product was dissolved in acetone, reprecipitated with H_2O, dried and weighed. For H_2O-soluble side-chains (starch - polyacrylamide), 2 g of the copolymer was added to 90 ml of anhyd. acetic acid and 10 ml of 60% $HClO_4$ heated to 80°; after 20 min the reaction mixture was poured into H_2O - ethanol (1 : 1), and the precipitated product was filtered off, washed with ethanol, vacuum-dried and weighed. The percentage recovery of vinyl polymer had a standard deviation of 0·45, and was in agreement with results obtained by other methods and with the calculated value. The new procedure was rapid, gave complete hydrolysis, and did not lead to modification of the vinyl polymers. I. R. H. C.

1C88. **Determination of vinyl acetate as comonomer in polyacrylonitrile.** Stein, G.; and Saglam, M. (Textilforschungsanst., Krefeld, W. Germany). *Melliand Textilber.*, 1977, **38** (4), 337 (in German).—After purifying by extraction for 5 h with acetone and methanol, the comminuted fibre (100 mg) is heated in a sealed tube with toluene-*p*-sulphonic acid monohydrate (1·5 g) for 3 h at 155° to 160°. After cooling the products, aq. 0·2% propionic acid (as

internal standard) is added, and the resulting suspension is analysed for acetic acid by g.l.c. (at 170°) on a column of Porapak Q, with He as carrier gas (25 ml min^{-1}) and flame ionisation detection. L. A. O'N.

1C89. **Mass-spectrometric analysis of compounds formed by pyrolysis of polyacrylonitrile and its co-polymers.** Chaigneau, M. (Serv. Anal. Gaz, C.N.R.S., Paris, France). *Analusis*, 1977, **5** (5), 223–227 (in French).—The pyrolysis of polyacrylonitrile and of the copolymers of acrylonitrile with styrene with and without butadiene was studied at fixed temp. between 500° and 1000° and also with the temp. increasing from 245°. The compositions and amounts of the products are tabulated. B. D. H.

1C90. **Bibliographical study on analysis of di-isocyanates.** Valero, F. *Bull. Lab. Prof.*, 1976, (43), 1–23 (in French).—A review is presented of methods (based on, *e.g.*, distillation, g.l.c., column chromatography, colorimetry, spectrophotometry, and n.m.r. spectroscopy) for the determination of volatile monomeric di-isocyanates in adducts or pre-polymers and in the atmosphere. Determination of isomers, and of one di-isocyanate in the presence of others, is discussed, as well as specific methods for hexamethylenedi-isocyanate and 'diphenylmethane di-isocyanate'. (45 references.)

World Surf. Coat. Abstr.

1C91. **Investigation of composition of polyurethans by pyrolysis - gas chromatography.** Zorina, N. I.; Tsarfin, Ya. A.; and Karnishin, A. A. (All-Union Sci.-Res. Inst. Synth. Resins, Vladimir, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1190–1194 (in Russian).—For the identification of polyhydric alcohols, the sample (≃1 mg) was pyrolysed on a Ni - Cr filament at 300° to 500° for 5 s. The pyrolysis products were swept by Ar into a glass column (70 cm × 4 mm) packed with 15% of polyoxyethylene glycol 4000 on Chromosorb W and operated at 120°, with Ar as carrier gas and flame ionisation detection. The method was suitable for separating and identifying ethanediol, propane-1,2-diol, butane-1,4-diol and ϵ-caprolactone as the major products; minor components of the pyrolysates were acrylaldehyde, acetonitrile, acrylonitrile, benzene, acetone and ethyl methyl ketone. M. P.

1C92. **Polarographic determination of cumene hydroperoxide and dicumyl peroxide in the presence of each other in industrial mixtures.** Gregorowicz, Zbigniew; Cebula, Jan; and Gorka, Piotr (Inst. Gen. Anal. Chem., Silesian Polytech., Gliwice, Poland). *Fresenius' Z. Anal. Chem.*, 1977, **284** (4), 287–288 (in German).—Cumene hydroperoxide (**I**) and dicumyl peroxide (**II**) were determined in the presence of each other and of acetophenone, acetone, α-methylstyrene, cumene, phenol, H_2O and αα-dimethylbenzyl alcohol in mixtures from the production of plastics. Samples (1 ml) were taken from the reaction vessel at 30-min intervals and filtered, and a weighed amount (containing 2 to 28 mg of **I** or 5 to 40 mg of **II**) was made up to 10 ml with 96% ethanol; 1-ml portions of this soln. were used for polarography. For the determination of **I**, the supporting electrolyte was 5 ml of a soln. (in 95% ethanol) 0·3M in LiCl and 0·1M in KCl; for **II**, the electrolyte was 5 ml of satd. tetramethylammonium chloride soln. in dimethylformamide, and triphenylphosphine (100 mg) was added to reduce **I**, which interfered in the determination of **II**. Hydrogen was removed within 10 min, 0·3 ml of 0·25% gelatin soln. was added, and N was bubbled through the cell for 1 min before the polarogram was recorded. Tolerance levels for the other sample components are reported. The maximum relative error in determining **I** or **II** was ±5%. C. K. L.

1C93. **Analysis of epoxy-resins by gel-permeation chromatography.** Schulz, G.; and Raubach, H. (Zentralinst. Org. Chem., Akad. Wiss. DDR, Berlin, E. Germany). *Plaste Kautsch.*, 1977, **24** (5), 325–328 (in German).—As an aid to identification, the separation of a range of epoxy-resins has been carried out on columns of cross-linked polystyrene, with dimethylformamide as solvent; peaks were identified by m.s. of appropriate fractions. Resins examined were the reaction products of bisphenol A (4,4′-isopropylidenediphenol) or its tetrabromo-derivative with 1-chloro-2,3-epoxypropane or the diglycidyl ether of bisphenol A. Satisfactory calibration graphs of mol. wt. *vs.* 'count number' were obtained for the various series of oligomers, but some anomalies of separation were encountered with molecules containing phenolic end-groups. L. A. O'N.

1C94. **Determination of oligomers in epoxy-resins from bisphenol A [4,4′-isopropylidenediphenol] and epichlorhydrin [1-chloro-2,3-epoxypropane].** Braun, D.; and Lee, D. W. (Dtsch. Kunststoff-Inst., Darmstadt, W. Germany). *Angew. Makromolek. Chem.*, 1977, **57** (892), 111–122 (in German).—Pure oligomers obtained by preparative gel-permeation chromatography (g.p.c.) were used to establish calibration graphs for use in the quantitative characterisation of such resins prepared with different reactant ratios by analytical g.p.c. (Merckogel OR 6000 columns; tetrahydrofuran solvent; u.v. detection). Epoxy-resins with small amounts of by-products showed distributions corresponding to those calculated theoretically; resins with larger amounts of incompletely epoxidised components showed appreciable deviations. L. A. O'N.

1C95. **Automated identification of automobile lacquers and paints.** Apostolov, D.; and Kal'pova, I. *Yad. Energ.*, 1976, **4** (4), 92–96 (in Bulgarian). *Ref. Zh., Khim.*, 19GD, 1977, (17), Abstr. No. 17G223.

1C96. **Identification of automobile paints by laser-probe mass spectrometry.** Brochard, G.; and Eloy, J. F. (Lab. Inter-reg. Police Scient., Lyon, France). *Analusis*, 1977, **5** (5), 242–248 (in French).—The technique described previously by Eloy (*Anal. Abstr.*, 1971, **21**, 3971) has been applied to individual flakes of paint. The variation of composition with depth can be plotted as a gradient profile for specific elements in the sample, enabling successive layers of paint to be distinguished at thicknesses of up to 70 μm. B. D. H.

1C97. **Long-chain phenols. VIII. Quantitative analysis of unsaturated constituents of phenolic lipids by thin-layer chromatography - mass spectrometry.** Tyman, J. H. P. (Sch. Chem., Brunel Univ., Uxbridge, Middx., England). *J. Chromatogr.*, 1977, **136** (2), 289–300.—The method was applied to cashew-nut shell liquid, the saturated and mono-, di- and tri-ene components of each phenol being separated by t.l.c. on silica gel - $AgNO_3$ with, *e.g.*, ammoniacal ethyl acetate - $CHCl_3$ (1 : 19) as mobile phase. The bands, made visible with Rhodamine 6G (C.I. Basic Red 1), were extracted with ethyl ether - methanol (9 : 1) containing 0·1% of BHT. The mass spectra of the individual constituents of each component phenol and of the relevant standard

mixture were recorded and peak heights were measured. The results compared well with those obtained by g.l.c. G. P. C.

1C98. **Direct micro-determination of lead in wax crayons by flameless atomic-absorption technique.** Castellani, Franca; Riccioni, Rosilio; Gusteri, Maura; Bartocci, Vito; and Cescon, Paolo (Ist. Chim., Univ. Camerino, Italy). *At. Absorpt. Newsl.*, 1977, **16** (3), 57–59.—Lead was determined in wax crayons by flameless a.a.s. at the Pb 283·3-nm line. Two procedures were used; either a solid sample (0·15 mg) was inserted into the furnace, dried at 100° for 60 s, charred at 500° for 120 s and atomised at 2300° for 10 s with gas flow, or, alternatively, a 1-g sample was charred in a muffle-furnace at 500°, the residue was treated with HNO_3 and the vol. was made up to 100 ml with H_2O, and 10-μl portions of the supernatant liquid were injected into the furnace and analysed with use of the same heating program as before. The two procedures gave closely similar results, which are compared with analyses by differential pulse polarography. Concentrations of Pb ranged from 9·3 p.p.m. in yellow crayons to 31 p.p.m. in blue ones. The coeff. of variation was $\simeq$1% for each colour (10 determinations).
C. K. L.

1C99. **Luminescence analysis of ingredients of rubber mixtures.** Ural'skii, M. L.; Makarova, A.; Gorelik, R. A.; Tikhonov, G. P.; and Bukanov, A. M. *Proizved. Shin Rezinotekh. Asbestotekh. Izd.*, 1976, (10), 24–27 (in Russian).—The luminescence of the individual ingredients was excited by the filtered 360-nm radiation of a high-pressure mercury lamp and studied visually, also by means of a luminescence microscope and a luminescence spectrometer. The number of substances studied and listed in the paper in various categories were—vulcanisation activators 8, pre-vulcanisation inhibitors 7, vulcanisation agents and accelerators 13, physical preservatives 5, chemical preservatives 8, and plasticisers and fillers 10. The self colours and luminescence colours are tabulated together with the relative integral luminescence intensities as percentages calculated from the area under the spectral curves (examples are shown). The method can be used for (*i*) routine control of purity, (*ii*) detecting heterogeneity in mixtures and manufactured parts, and (*iii*) studying surface migration. G. S. S.

1C100. **Spectrophotometric determination of peroxides in hydrocarbons and latex.** Kirsanova, L. Kh.; Surpina, D. E.; and Rapoport, E. L. (Synthetic Rubber Works, Kuibyshev, USSR). *Prom-st. Sint. Kauch.*, 1976, (11), 11–12 (in Russian).—For hydrocarbons, mix a 1 to 2-g sample with sufficient acetic acid - $CHCl_3$ (2 : 1) to make the total vol. 25 ml, then de-aerate the soln. with a stream of Ar and add 1 ml of 50% KI soln. After keeping the soln. in the dark for 30 min, measure the absorbance at 320, 360 and 440 nm; the concn. of peroxides is calculated from a given mathematical expression. For latex, coagulate the sample (1 to 2 ml) by adding it dropwise to 50 ml of ethanol and then analyse a 2-ml portion of the soln. by the method described.
A. S. W.

1C101. **Determination of calcium stearate in rubber.** Rapoport, E. L.; Lopatina, I. V.; Shmeleva, L. Ya.; and Konoshchenok, T. V. (Synthetic Rubber Works, Kuibyshev, USSR). *Prom-st. Sint. Kauch.*, 1976, (11), 13–14 (in Russian).—Mix 3 to 5 g of the finely shredded sample with 100 ml of ethanol - toluene (3 : 7), and add 1 drop of conc. H_2SO_4. Reflux for 30 min, cool and add 60 ml of ethanol. Shake vigorously to separate the polymer, transfer the soln. to a titration cell, and titrate potentiometrically with ethanolic 0·1M-KOH. The titration curve has two inflections, the first corresponding to the titration of H_2SO_4, the second to stearic acid. The method is simple, rapid and suitable for the range 0·5 to 1·5%. The coeff. of variation is $\simeq$5%.
A. S. W.

1C102. **Determination of derivatives of piperidine-1-carbodithioic acid by thin-layer chromatography.** Grushevskaya, N. Yu.; and Kazarinova, N. F. (All-Union Sci.-Res. Inst. Hyg. and Toxicol. Pesticides, Polymers and Plastics, Kiev, USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 1036–1038 (in Russian).—Vulcanisation accelerators such as Zn piperidine-1-carbodithioate (**I**), dipentamethylenethiuram tetrasulphide (Tetrone A) (**II**), bis(piperidinothiocarbonyl) disulphide (**III**) and piperidinium piperidine-1-carbodithioate (**IV**) were separated and determined on a layer of silica gel - gypsum, with benzene - CCl_4 (9 : 1) as mobile phase. After drying the chromatogram at ambient temp., the zones were detected by spraying with aq. 3% $CuSO_4$, Dragendorff reagent or 0·05% dithizone soln. in CCl_4. Graphs of log. wt. of compound in the zone (in μg) *vs.* square root of zone area were rectilinear for 2 to 15 μg of **II**, **III** and **IV** and 1 to 16 μg of **I**. Traces (>0·025 p.p.m.) of all the compounds in water samples could be determined by this method after extraction into $CHCl_3$. M. P.

1C103. **Determination of deoxyribose by colorimetry: method of differentiating between free and constituent sugar in deoxyribonucleic acid, and its application to the analysis of gelatin.** Pouradier, J. (Centre Rech., Kodak-Pathe, Vincennes, France). *Bull. Soc. Chim. Fr., I*, 1977, (5–6), 455–456 (in French).—The method is based on the selective effect of trichloroacetic acid (**I**) on the extraction into $CHCl_3$ of coloured products of the reaction of indole with deoxyribose (**II**). In the presence of 0·1M-**I**, >95% of the free sugar is extracted and only $\simeq$10% of the **II** associated with DNA. The gelatin sample is hydrolysed with HCl at room temp., and the soln. is then heated with 0·04% indole soln. at $\simeq$95° for 10 min; interfering coloured products (due to, *e.g.*, arabinose) in the cool soln. are extracted (×3) into $CHCl_3$, and the absorbance of the aq. phase is measured at 489 nm to obtain the total concn. of **II**. To the aq. phase is then added 0·1M-**I**, the extraction with $CHCl_3$ is repeated to separate reaction products of free **II**, and the absorbance of the aq. phase is again measured at 489 nm to determine the **II** associated with DNA. The concn. of free **II** is obtained by difference. The calibration graph is prepared with use of the gelatin sample plus added amounts (0·1 to 1 mg) of **II** and DNA. Tissue samples must be hydrolysed under more severe conditions than are used for gelatin.
W. J. B.

1C104. **Analysis of explosives by high-performance liquid chromatography and chemical-ionisation mass spectrometry.** Vouros, Paul; Petersen, B. A.; Colwell, L.; Karger, B. L.; and Harris, Howard (Inst. Chem. Anal., Appl. Forensic Sci., Northeastern Univ., Boston, Mass. 02115, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1039–1044.—Sample extracts are separated on a column (25 cm × 3·1 mm) of Partisil (10 μm), with a mobile phase (*e.g.*, 1,2-dichloroethane) depending on the mixture and a flow

rate of 0·5 ml min^{-1}. Mass spectra are obtained with NH_3 as reagent gas. The method has been applied to the analysis of simple residues from test explosions. G. P. C.

1C105. **Identification of individual polychlorinated biphenyls in a mixture by gas - liquid chromatography.** Albro, Phillip W.; Haseman, Joseph K.; Clemmer, Thomas A.; and Corbett, Barbara J. (Natl. Inst. Environ. Health Sci., Research Triangle Park, N.C. 27709, U.S.A.). *J. Chromatogr.*, 1977, **136** (1), 147–153.—Retention indices were computed for the 210 possible chlorinated biphenyl isomers on 13 g.l.c. liquid phases from the data obtained from 38 isomers. All possible paired comparisons of retention indices were made for each pair and each set of three liquid phases. Based on a retention-index difference of 10, it was deduced that five columns are needed to distinguish all the isomers. Consideration of column efficiencies, analysis time, separation and availability led to the selection of several liquid phases for qualitative and quantitative determination of chlorinated biphenyl isomers in commercial samples. M. J. Sl.

See also Abstracts: **1A1**, Determination of impurity elements: review. **1A3**, Various reagents for organic species: review. **1A7**, 'Chemical masking' in organic analysis. **1B5**, Analysis of coal fly ash. **1B116**, Determination of organic impurities in tetrachlorosilane. **1B140**, Study of reactions of nitrosamines. **1B143**, Determination of N and C. **1B145**, Formation of derivatives for g.l.c. of organic acids. **1B216**, Determination of thiourea. **1B241**, Determination of Pt and Ir in reforming catalysts. **1D21**, Determination of N in organic materials. **1D92**, Determination of cyclohexanone. **1D99**, Determination of maltose. **1D103**, Freeze-drying isolation of organic acids. **1D114**, Analysis of linseed oil. **1D138**, Reagent for amino-compounds. **1E12**, Determination of organic substances. **1E13**, H.p.l.c. of dyes. **1E34**, Analysis of essential oils. **1E57**, Determination of aromatic amines. **1F14**, Determination of phthalate esters. **1F15**, Determination of methyltin compounds. **1F22**, Improved reaction for sugars. **1F53**, Analysis of fusel oils. **1G13**, Determination of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin. **1G20**, Chemistry of ethylenethiourea. **1H2**, Sampling of cotton dust. **1H9**, Determination of HCN in fire gases. **1H11**, G.c. - m.s. of organic substances. **1H13**, Monitoring of vinyl chloride. **1H16**, Determination of organomercury compounds. **1H33**, Determination of organic C. **1H43** to **1H51**, **1H58**, Determination of organic compounds in water. **1H59**, Determination of xylenols in waste water. **1J7**, Apparatus for isolation of organic chemicals. **1J9**, Apparatus for determining N in organic compounds. **1J11**, Material for column separation of sugars. **1J13**, Materials for sorption of metals from organic solutions. **1J19**, Detector for organic S compounds. **1J27**, T.l.c. of organic compounds labelled with ^3H. **1J31**, Chromatograph for analysis of compounds labelled with ^{11}C and ^{35}S. **1J34**, Subtractive g.c. of organic compounds. **1J38**, Detector for g.c. of N-containing organic compounds. **1J39**, Fluorescence detection in g.c. of polycyclic hydrocarbons. **1J40**, Detector for Br compounds. **1J41**, Detector for Si compounds. **1J86**, Fluorescence analysis of aromatic compounds. **1J90**, Raman spectrometry of organic compounds. **1J94**, **1J96**, M.s. of organic compounds. **1J104**, Electrolytic chromatography of oxalate. **1J106**, E.m.f. values for acetic acid.

D—BIOCHEMISTRY

General; elements (including toxic metals)

1D1. **Preservation of the plasma ash-pattern of biological specimens by subsequent coating with a plasma-polymerised thin film.** Tsuji, Osamu; Wydeven, Theodore; and Hozumi, Keiichiro (NASA–Ames Res. Center, Moffett Field, Calif. 94035, U.S.A.). *Microchem. J.*, 1977, **22** (2), 229–235.—The specimen (10 μm thick), supported by a glass slide, is incinerated in a plasma reactor by means of an O glow discharge (13·6 MHz). The residual ash is then coated with a plasma-polymerised film ($\simeq$4000 Å) of perfluorobut-2-ene in the same reactor, so that the film is deposited on the ash directly from the vapour phase. The coated ash is mechanically strong and non-hygroscopic in ambient air, so that specimens can be stored for at least 60 days. The flow system for ashing and coating is shown diagrammatically. W. J. B.

1D2. **Chromatographic and other assay approaches.** Reid, Eric (Wolfson Bioanal. Centre, Univ. Surrey, Guildford, England). *Methodol. Dev. Biochem.*, 1976, **5**, 55–70.—A review is presented, with special reference to the analysis of biological fluids; the topics discussed include h.p.l.c., g.c. and t.l.c. Ways of ensuring optimum conditions are summarised, and sample-preparation procedures are considered. (47 references.) B. H.

1D3. **Use of isotachophoresis for analysis of muscle extracts.** Gower, David C.; and Woledge, Roger C. (Dept. Physiol., University Coll., Gower St., London, W.C.1, England). *Sci. Tools*, 1977, **24** (2), 17–21.—Aqueous soln. of freeze-dried methanolic extracts of frog sartorius muscles were subjected to isotachophoresis in a PTFE tube (43 cm × 0·5 mm) at 15°. The leading electrolyte was 5mM-HCl, containing β-alanine (18mM) to provide the counterion, in aq. 0·5% methylcellulose at pH 3·92. The terminating electrolyte was 5mM-hexanoic acid. The separated zones were detected by u.v. absorption at 254 nm, and non-u.v.-absorbing material was estimated from the distance between the u.v.-absorbing zones. The integrated areas under the curves on the chart recording, after conversion into absorbance, are rectilinearly related to the amounts of material in the respective zones. Good agreement with other methods was found for ATP (u.v.-absorbing) and phosphocreatine (non-u.v.-absorbing). H. K. M.

1D4. **Analysis of urinary calculi: methods of study.** Ligabue, A.; and Biagi, R. (Lab. Anal. Clin., Osp. 'M. Malpighi', Bologna, Italy). *Quad. Sclavo Diagn. Clin. Lab.*, 1976, **12** (4), 501–513 (in Italian).—For i.r. analysis, the sample is washed with water, ethanol and ethyl ether, then weighed and ground in an agate mortar. A dry 3-mg portion is ground with 600 mg of KBr, and 150 mg of the mixture is compressed at 10 t and examined by spectrophotometry between 4000 and 625 cm^{-1}. For qualitative chemical analysis, 10 to 20 mg of the pulverised material is treated with 0·5 ml of 1·7M-HCl, and the soln. is tested for carbonate, cystine, phosphate, Mg, Ca, NH_4^+, uric acid and oxalic acid. In the investigation of 100 samples, the chemical method frequently gave falsely positive results for phosphate, Mg, Ca, NH_4^+, uric acid and oxalic acid, and gave

falsely negative results for uric and oxalic acids in some instances; in addition, the chemical method did not indicate the type of phosphate. The i.r. method gave reliable results and was rapid (a test took 5 min). L. B.

1D5. **Simultaneous determination of nitrogen, magnesium, silicon, phosphorus and potassium in plant material by 14-MeV neutron-activation analysis.** Segebade, C. (Bundesanst. Materialpruefung, Berlin, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 23–29 (in German).—The dried powdered sample [≃3 g, in a polyethylene capsule (2·6 cm diam. × 8·3 mm deep) with aluminium discs of the same diam. on both sides to act as flux monitors] was irradiated with generator-produced neutrons for 10 min at a flux of 10^9 n cm^{-2} s^{-1} and then transferred to a test-tube, and its γ-spectrum was recorded with a NaI(Tl) detector connected to a 1024-channel pulse-height analyser and was evaluated with a 16K computer. As standard, a mixture of $Mg_2P_2O_7$, KSCN, SiO_2 and cellulose (containing 0·57, 0·92, 1·48, 1·17 and 1·59% of N, Mg, Si, P and K, respectively) was irradiated and evaluated similarly. A diagram of the irradiation and transportation system is presented. Satisfactory results were obtained when the elements were determined in NBS standard orchard leaves; coeff. of variation were ≃14 to 16%. H. L. L.

1D6. **Studies on heavy-metal content of tobacco products and tobacco smoke.** Franzke, C.; Ruick, G.; and Schmidt, M. (Sekt. Chem., Humboldt Univ., Berlin, E. Germany). *Nahrung*, 1977, **21** (5), 417–428 (in German).—Cigarette tobacco is boiled under reflux with HNO_3 - $HClO_4$ (4:1), residual $HClO_4$ is evaporated by heating on a hot-plate, the residue is diluted with M-H_3PO_4 - 0·5M-$HClO_4$, and Cu, Pb, Cd and Zn are determined by square-wave polarography at −0·02, −0·40, −0·60 and −1·00 V, respectively, and the method of standard addition. The recoveries were 101·8, 98·2, 94·9 and 92·3%, respectively, and the corresponding coeff. of variation were 7·1, 4·7, 3·3 and 1·1%. Cigarettes are smoked in a smoking machine, and the smoke is absorbed in the HNO_3 - $HClO_4$; the determination is continued as for tobacco. R. F. S.

1D7. **Computerisation of the gas-phase analysis of cigarette smoke.** Sloan, C. H.; Lewis, J. S.; and Morie, G. P. (Tennessee Eastman Co. Div., Eastman Kodak Co., Kingsport, Tenn. 37662, U.S.A.). *Tob. Sci.*, 1977, **21**, 57; *Tobacco*, 1977, **179** (9), 33.—Analyses were effected on a g.c. column (12 ft. × 0·125 in.) containing Porapak Q and interfaced with a Hewlett-Packard 3352B laboratory data system that was used to calculate the peak areas and hence the relative amounts of cigarette-smoke components. The software included a program (in BASIC) that enabled the system to store data for control and test filter-tipped cigarettes and to calculate the percentage decrease in 22 specific vapour-phase components. Data for replicate analyses could be stored and their average values used in subsequent calculations. Chem. Abstr.

1D8. **Chemical studies on tobacco smoke. XLIX. Gas-chromatographic determination of hydrogen cyanide and cyanogen in tobacco smoke.** Brunnemann, Klaus D.; Yu, Louis; and Hoffmann, Dietrich (Div. Environ. Carcinogenesis, Naylor Dana Inst. Disease Prevention, Valhalla, N.Y. 10595, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (1), 38–42.—Smoke is passed through 0·1M-NaOH to absorb HCN, and the absorbent is shaken with M-NaH_2PO_4, chloramine T soln. and hexane; this converts the HCN into CNCl, which is determined by g.l.c. (at 55°) of the hexane layer on a column (4·5 m × 2 mm) of 7% of Hallcomid M-18 on Gas-Chrom Q (80 to 100 mesh), with Ar - CH_4 (19:1) as carrier gas (40 ml min^{-1}) and ^{63}Ni electron-capture detection; the retention time of CNCl is 1·6 min, and the response is rectilinear up to 5 μg ml^{-1} in the soln. injected. To determine $(CN)_2$, the smoke is collected in a PTFE bag and analysed by g.l.c. at 40° on a stainless-steel column (40 m × 3 mm o.d.) packed with 20% of triacetin on Chromosorb P NAW (60 to 80 mesh), with He (30 ml min^{-1}) as carrier gas; $(CN)_2$ was eluted in ≃27 min. Results for side-stream and main-stream smokes are discussed. B. H.

1D9. **Evaluation of the horseradish peroxidase - scopoletin method for measurement of hydrogen peroxide formation in biological systems.** Boveris, A.; Martino, E.; and Stoppani, A. O. M. (Inst. Quim. Biol., Fac. Med., Buenos Aires, Argentina). *Anal. Biochem.*, 1977, **80** (1), 145–158.—The glucose oxidase system was used as a source of H_2O_2 to assess the accuracy and reliability of the method cited. The assay was carried out in an Aminco-Bowman spectrofluorimeter with excitation and emission wavelengths of 365 and 450 nm, respectively, and the optimum concn. of horseradish peroxidase and of scopoletin (in the presence of catalase) were determined. The method was applied to various mitochondrial preparations including rat and pigeon heart, rat liver, mung bean and yeast, and in all instances the results were 2 to 60% lower than those obtained with the cytochrome peroxidase method (Boveris *et al., Biochem. J.*, 1972, **128**, 617). Correction for catalase interference can be made. R. A. H.

1D10. **Spectrographic determination of calcium, barium and strontium in biological samples, food products and water.** Khotomlyanskaya, G. Ya.; Tomilin, Yu. A.; and Golovchenko, M. G. (Nikolaev Reg. Health Authority Sta., USSR). *Gig. Sanit.*, 1976, (11), 100–101 (in Russian).—The sample soln. was placed on a flat-topped (pre-ignited) carbon electrode after the electrode had been treated with 3% polystyrene soln. in benzene. The upper electrode was a carbon rod with a 120° point. Both electrodes were pre-ignited in an a.c. arc at 8 A for 30 s. A soln. of $Co(NO_3)_2$ was used as internal standard. The concn. of Ca, Ba and Sr and the pH were matched against prepared standards. The analytical line pairs used were: Ca I 3180·52 - Co I 3044·01, Ba I 3071·59 - Co I 3044·01 and Sr II 3380·71 - Co I 3044·01. M. R. M.

1D11. **Effect of different sample preparation methods on atomic-absorption spectrophotometric determination of calcium in plant material.** Adrian, William J.; and Stevens, Marilyn L. (Div. Wildlife, Res. Center, Fort Collins, Colo. 80522, U.S.A.). *Analyst* (*London*), 1977, **102** (1215), 446–452.—Detailed results are presented of Ca determinations on NBS standard orchard leaves (certified value 2·09% of Ca), with use of two dry-ashing and three wet-ashing procedures, and with La or Sr as releasing agents. Sample preparation procedure had a considerable effect on the result obtained, but satisfactory values could be achieved when a N_2O - acetylene flame was used in conjunction with a test soln. diluted to contain 1 to 5 p.p.m. of Ca. R. W.

1D12. **Liquid scintillation counting of calcium-45 in plant and soil material.** Waller, S. S.; and Dodd, J. D. (Dept. Range Sci., Texas A & M Univ., College Station, Tex. 77843, U.S.A.). *Health Phys.*, 1977, **32** (3), 185–187.—Plant material was dried, ground and heated to a uniform ash, to which was added H_2O and 0·57N-HCl, and the resulting soln. was kept in the dark for 1 h before counting. Soil samples were air-dried, ground, oven-dried and placed in a column containing a glass-fibre plug; the soil was extracted with 0·23N-$MgCl_2$, and the extract was counted in the same manner as for the plant samples. Counting was performed with use of a Beckman LS 100 liquid scintillation counter and Beckman Ready Solv TM Solution XI solvent system as scintillator. Extraction efficiencies were 85·4 to 95·1% and 95·3 to 97·1% for plant and soil samples, respectively, and the activities were 50 to 200 nCi g^{-1} (plants) and 24 nCi g^{-1} (soil).
CHEM. ABSTR.

1D13. **Determination of ionised calcium in blood serum.** Talakin, Yu. N.; and Kolomiets, V. V. (M. Gor'kii Med. Inst., Donetsk, Ukr. SSR). *Gig. Sanit.*, 1976, (11), 77–78 (in Russian).—An ion-selective electrode method was used to measure the active Ca^{2+} in blood serum at 25° ± 0·2° and pH 7·4. The presence of Hg alters the Ca balance and reduces the active Ca^{2+} level. Investigations on the effect of Hg levels from 0·001 to 0·1 μg l^{-1} are described.
M. R. M.

1D14. **Atomic-emission spectrometer/spectrograph for determination of barium in micro amounts of diatom ash.** Bankston, Donald C.; and Fisher, Nicholas S. (Dept. Chem., Woods Hole Oceanogr. Inst., Mass. 02543, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1017–1023.—The sample (<10 mg) of ash is mixed with pure SiO_2 to give a total wt. of 10 mg, the mixture is fused with 70 mg of $LiBO_2$, and the melt is poured into 7·5 ml of 4% HNO_3. This soln. is examined by means of a Spectrospan II-B single-channel spectrometer having an echelle diffraction grating and a d.c. Ar-plasma arc source, emission measurement being made at 455·4 nm. Down to 2 μg l^{-1} of Ba (≡2 p.p.m. of Ba in the ash) could be detected.
R. W.

1D15. **Determination of traces of cadmium by atomic absorption after extraction with dipivaloylmethane [2,2,6,6-tetramethylheptane-3,5-dione]. Possible application to cadmium in urine.** Lekehal, N.; Hanocq, M.; and Helson-Cambier, M. (Lab. Chim. Anal. et Toxicol., Inst. Pharm., Univ. Libre, Bruxelles, Belgium). *J. Pharm. Belg.*, 1977, **32** (1), 76–88 (in French).—The procedure described previously for the determination of Mn (*Anal. Abstr.*, 1976, **31**, 6D11) has been adapted to the determination of Cd either by flameless a.a.s. or by a.a.s. with use of an air - acetylene flame. Beer's law is obeyed for 5 to 500 p.p.b. of Cd in the sample (water or urine) or in the soln. obtained after mineralisation of the urine sample.
S. M. M.

1D16. **Trace chemistry of toxic metals in biomatrices. II. Voltammetric determination of traces of cadmium and other toxic metals in human whole blood.** Valenta, P.; Ruetzel, H.; Nuernberg, H. W.; and Stoeppler, M. [Inst. Chem., Inst. 4, Appl. Phys. Chem., Nuclear Res. Centre (KFA), Juelich, W. Germany]. *Fresenius' Z. Anal. Chem.*, 1977, **285** (1), 25–34.—The application of differential-pulse anodic-stripping voltammetry to determinations of trace metals in blood is described. Samples are ashed in O at 200°, followed by dissolution of the ash in 0·1M-HCl; the pH is then adjusted to 2·0 to 2·4 with NaOH. Possible sources of contamination by Pb and Cd are suggested, and methods for minimising their effect are proposed. Detection limits for Pb (0·5 p.p.b.) and Cd (0·01 p.p.b.) with coeff. of variation of ±5% and ±20%, respectively, are reported.
J. K. C.

1D17. **Modified method for determining mercury in biological samples by atomic-absorption spectrophotometry.** Szprengier, Teresa (Zakl. Farmakol. Toksykol., Inst. Weter., Pulawy, Poland). *Med. Weter.*, 1977, **33** (3), 182–184 (in Polish).—The method is a variant of that of Zmudzki and Szprengier (*Ibid.*, 1973, **29**, 120). The wet-ashed sample is treated with a reductant containing, per 100 ml, 25 g of $SnCl_2$, 5 g of $CdCl_2$ and 20 ml of M-HCl; the mixture is then aerated, and the Hg vapour is determined by a.a.s.; the sensitivity is 1 p.p.b.
CHEM. ABSTR.

1D18. **Automated method for determination of mercury in urine.** Richardson, Ralph Alan (Dept. Clin. Chem., Auckland Hosp., Park Rd., Auckland, New Zealand). *Clin. Chem. (Winston-Salem, N.C.)*, 1976, **22** (10), 1604–1607.—After conventional reduction of Hg to the element with $SnCl_2$ soln. in an enclosed system constructed from AutoAnalyzer components, the Hg vapour is entrained in a stream of air or N for flameless a.a.s. at 253·7 nm. Twenty 0·5-ml samples can be processed in 1 h, and the detection limit is 2 μg l^{-1} of Hg. There is no interference from Pb (up to 5 mg l^{-1}), nor from As at levels likely to be present in urine.
B. H.

1D19. **Determination of carbon monoxide by means of a palladium - promazine complex.** Jones, June K. (Dept. Toxicol. and Criminal Invest., Auburn, Ala. 36830, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (1), 54–56.—In the central well of a Conway cell is placed 2·5mM-$PdCl_2$, and in the outer compartment are placed the blood sample and the CO-liberating reagent [acetic acid (10 ml) plus H_2O (90 ml) saturated with Pb acetate and containing 1% (v/v) of Triton X-100]. After diffusion for 4 h, an aliquot of the Pd soln. is treated with Na acetate - HCl buffer soln. (pH 2) and 0·06M-promazine hydrochloride, and the absorbance at 578 nm is measured; this value is inverseiy proportional to the CO content in the blood up to 30% (v/v).
B. H.

1D20. **Determination of lead in biological samples by atomic-absorption spectrophotometry.** Zmudzki, Jan (Zakl. Farmakol. Toksykol., Inst. Weter., Pulawy, Poland). *Med. Weter.*, 1977, **33** (3), 179–181 (in Polish).—The method is similar to that of Yeager *et al.* (*Anal. Abstr.*, 1972, **23**, 512). The dry-ashed sample is dissolved in M-HCl or oxalic acid (1 g of acid per g of ash), and Pb is extracted by consecutive treatment of the soln. with soln. of phenol red, ammonium citrate, hydroxylamine (to give a red colour), KCN and ammonium pyrrolidine-1-carbodithioate and with isobutyl methyl ketone. The Pb in the organic phase is determined by a.a.s. at 217 nm in an acetylene - air flame. Average recovery of 0·1 to 0·5 p.p.m. of Pb ranged from 91 to 98%, and the sensitivity was 5 p.p.b. The coeff. of variation ranged from 2·97% for 1 p.p.m. of Pb to 13·19% for 0·1 p.p.m.
CHEM. ABSTR.

1D21. **Simple procedure for mass-spectrometric micro-analysis of nitrogen in particulate organic matter with special reference to nitrogen-15 tracer**

experiments. Wada, Eitaro; Tsuji, Takashi; Saino, Toshiro; and Hattori, Akihiko (Ocean Res. Inst., Univ., Nakano, Tokyo, Japan). *Anal. Biochem.*, 1977, **80** (1), 312–318.—Particulate organic matter from, *e.g.*, marine organisms was collected on glass-fibre filters (Whatman type C) and this was decomposed to N by a modified Dumas method with use of a catalyst-packed vessel consisting of three concentric silica tubes (shown diagrammatically). The N produced was purified, collected on a molecular sieve and introduced into the mass spectrometer for determination of the ^{15}N content. Recovery of N from Na glutamate was 96·4 ± 1·9% at the level of 2·5 to 20 μg-atoms of N. R. A. H.

1D22. **Determination of nitrogen-15 by emission spectrometry: procedure for use with small amounts of nitrogen.** Lloyd-Jones, C. P.; Adam, J. S.; Hudd, G. A.; and Hill-Cottingham, D. G. (Univ. Bristol, Dept. Agric. and Hortic., Long Ashton, Bristol, England). *Analyst* (*London*), 1977, **102** (1215), 473–476.—Digest up to 150 mg of plant material (containing 20 to 500 μg of N) with 2 ml of 0·5% Se in conc. H_2SO_4 and dilute the cooled digest to 20 ml. If NO_3^- are present, digest the sample with 5% salicylic acid in H_2SO_4, and, if Cu or Hg was used as Kjeldahl catalyst, treat the soln. with H_2S and remove the ppt. After diluting a 2-ml aliquot with 9 ml of M-NaOH, determine total N with an NH_4^+-selective electrode. Neutralise the remainder of the digest with 10M-NaOH, then adjust until 0·4M in NaOH and heat on a water-bath with 4 ml of Nessler reagent. Centrifuge and reject the soln. Treat the ppt. with sufficient ethanolic HCl to give ≃250 μg of N per ml in the soln., decant the NH_4Cl soln. and fill a short length of glass tube with a vol. of soln. containing ≃10 μg of N, heat it to leave a film of NH_4Cl and complete the determination of ^{15}N by Dumas combustion and emission spectrometry as described by Lloyd-Jones *et al.* (*Anal. Abstr.*, 1975, **28**, 4D12). R. W.

1D23. **Some 1,2-diaminobenzene [o-phenylenediamine] derivatives as reagents for gas-chromatographic determination of selenium with an electron-capture detector.** Shimoishi, Yasuaki (Dept. Chem., Fac. Sci., Okayama Univ., Japan). *J. Chromatogr.*, 1977, **136** (1), 85–93.—Thirteen derivatives of 2,1,3-selenadiazole were synthesised by reaction of *o*-phenylenediamine analogues with H_2SeO_3. By considering retention data, detector sensitivity and distribution between aq. soln. and toluene, 4,6-dibromobenzo-2,1,3-selenadiazole was considered to be the most suitable for the determination of Se. Selenium was determined in NBS bovine liver (50 or 100 mg) by digestion with HNO_3 (10 ml) until the vol. was reduced to 0·3 to 0·5 ml, and the N oxides were decomposed with M-urea (3 ml). To reduce Se^{VI} to Se^{IV}, conc. HCl (5 ml) was added, and the mixture was heated at 100° for 10 min. A 0·5-ml aliquot of the soln. was treated with 0·2% 3,5-dibromo-*o*-phenylenediamine in conc. HCl (2 ml), and the HCl concn. was made up to ≃M. The soln. was set aside for 2 h at room temp., and extracted with toluene (1 ml), which was washed with M-NaOH (4 ml) and 9M-HCl (2 ml). The organic phase was subjected to g.l.c. at 200° on a glass column (1 m × 4 mm) packed with 15% of SE-30 on Chromosorb W (60 to 80 mesh) and operated with N as carrier gas (34 ml min^{-1}) and a ^{63}Ni electron-capture detector. The limit of detection of Se in toluene was 1 ng ml^{-1}. M. J. Sʟ.

1D24. **Determination of selenium in biological samples by gas - liquid chromatography with electron-capture detection.** Poole, C. F.; Evans, Nancy J.; and Wibberley, D. G. (Dept. Pharm., Univ. Aston in Birmingham, England). *J. Chromatogr.*, 1977, **136** (1), 73–83.—Human blood, hair, urine and placenta, NBS bovine liver and orchard leaves (0·1 to 1 g) were digested with $Mg(NO_3)_2.6H_2O$ (4 g) and HNO_3 (10 ml) at 80° to 90° for 2 to 3 h. After evaporation to dryness and heating at 500° for 30 min, each residue was dissolved in HCl (8 ml) and heated at 100° for 10 to 15 min. The digest was transferred to a stoppered flask with 20% urea soln. (10 ml), 1% 4-nitro-*o*-phenylenediamine dihydrochloride soln. (2 ml) was added and the mixture was set aside for 4 h at room temp. The soln. was extracted with toluene (5 ml), and the organic phase (2 to 5 μl) was subjected to g.l.c. on a column (1·5 ft. × 0·25 in.) packed with 7% of OV-225 on Supasorb AW-HMDS (100 to 120 mesh) and operated at 210°, with N as carrier gas (35 ml min^{-1}) and electron-capture detection. The recovery of Se was 95 to 105%, and the limit of detection of 5-nitrobenzo-2,1,3-selenadiazole was 90 pg. A calibration graph prepared by adding 0·05 to 2 μg of Se to a HNO_3 - $Mg(NO_3)_2$ digest was rectilinear. M. J. Sʟ.

1D25. **Determination of fluorine in dental enamel by gamma-ray activation.** Raulo, J.; Bouchier, G.; and Jean, G. (Lab. Rech. Odontol., CEN, Saclay, France). *Actual. Odonto-Stomatol.*, 1976, (115), 579–588 (in French).—The γ-rays were produced by means of an electron beam from a linear accelerator and a platinum target, the max. beam energy being adjusted to 22 MeV, at an intensity of 100 μA, in order to minimise interference from other constituents of teeth, in particular Na. Pairs of teeth, in aluminium cartridges, were irradiated for 5 min, together with a standard of known F content, *e.g.*, LiF. The ^{18}F formed by the reaction $^{19}F(\gamma,n)^{18}F$ was determined by measuring, at 510 keV, the intensity of γ-rays produced by annihilation of the β^+-particles from ^{18}F, immediately after irradiation and then after 110 min (the half-life of ^{18}F). The detection limit was ≃10 p.p.m. of F. R. J. F.

1D26. **Measurement of fluoride in bone [biopsy samples].** Mernagh, J. R.; Harrison, J. E.; Hancock, R.; and McNeill, K. G. (Dept. Phys. and Med. and SLOWPOKE Reactor Facility, Univ. Toronto, Ont., Canada). *Int. J. Appl. Radiat. Isot.*, 1977, **28** (6), 581–583.—The sample (with or without formaldehyde soln.) in a sealed polyethylene tube was irradiated for 20 s in a flux of ≃10^{12} n cm^{-2} s^{-1}; the reactions $^{19}F(n,\gamma)^{20}F$ and $^{48}Ca(n,\gamma)^{49}Ca$ were induced, ^{20}F and ^{49}Ca were counted at 1·633 and 3·1 MeV, respectively, and the ratio of F to Ca was calculated. Solutions of $CaCO_3$ and NaF were used as standards, but were irradiated at a lower neutron flux. Timing had to be accurate to within 0·5 s. The detection limit was ≃50 μg of F. W. C. D.

1D27. **Direct atomic-absorption spectrometric determination of manganese in whole blood of unexposed individuals and exposed workers in a Norwegian manganese-alloy plant.** Tsalev, D. L.; Langmyhr, F. J.; and Gunderson, B. (Dept. Chem., Univ. Oslo, Norway). *Bull. Environ. Contam. Toxicol.*, 1977, **17** (6), 660–666.—Optimum experimental conditions for analysing whole blood were found to include use of a 2-μl sample with a heating programme of 90° for 40 s, 530° for 25 s, 1200° for 40 s, then atomisation at 2200° to 2250° for 3·5 s, measurements being

made at 279·5 nm. The limit of detection was 2 p.p.b. of Mn and concn. in the range 6 to 30 p.p.b. were measured. S. M. M.

Drugs and toxic compounds (including pesticides) in biological materials

1D28. **Compendium of therapeutic and toxic concentrations of toxicologically significant drugs in human biofluids.** Baselt, Randall C.; and Cravey, Robert H. (Dept. Lab. Med., Univ. Connecticut Health Center, Farmington, Conn. 06032, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (2), 81–103.—Data are tabulated on therapeutic concn. of 136 drugs and toxic concn. of 74 drugs in blood, plasma or serum. Where appropriate, concn. of physiologically active metabolites are also listed. Relevant pharmacological information is included. (238 references.) B. H.

1D29. **Sample preparation in high-performance liquid chromatography.** Dell, Dennis (Hoechst Pharm. Res. Lab., Milton Keynes, Bucks., England). *Methodol. Dev. Biochem.*, 1976, **5**, 131–134.—A review is presented of methods for clean-up before h.p.l.c. when determining drugs in biological fluids. Methods discussed include—removal of protein from plasma or serum by pptn., ultra-centrifugation or use of a pre-column; removal of lipids; and concentration of the drug and removal of inorganic salts from urine. (13 references.) B. H.

1D30. **Ion-pair solvent extraction and chromatography [in biochemical analysis].** Schill, Goran (Dept. Anal. Pharm. Chem., Univ. Uppsala, Sweden). *Methodol. Dev. Biochem.*, 1976, **5**, 87–102.—The mechanism of formation of ion-association complexes is discussed, and use of the principle in conjunction with solvent extraction, photometry, fluorimetry and partition chromatography is reviewed. Examples are given of the use of the technique in analysis of biological samples for basic drugs and their metabolites, carboxylic acids and sulphonamides, and glucuronide and sulphate conjugates of steroids. (22 references.) B. H.

1D31. **Solvent extraction [of drugs in biological samples].** Jones, C. R. (Dept. Drug Metab., Wellcome Res. Lab., Beckenham, Kent, England). *Methodol. Dev. Biochem.*, 1976, **5**, 107–113.—Techniques discussed for this purpose include (*i*) liquid - solid extraction (the solid being the freeze-dried sample or a mixture of blood with Na_2SO_4), and (*ii*) liquid - liquid extraction. Factors affecting the optimisation of conditions are considered. B. H.

1D32. **Quantitative thin-layer chromatographic determination of drugs in biological fluids.** Faber, D. B. (Lab. Toxicol. and Biopharm., Acad. Hosp., Free Univ., Amsterdam, Netherlands). *Methodol. Dev. Biochem.*, 1976, **5**, 39–44.—The advantages of t.l.c. for this purpose are reviewed, with particular reference to high-performance t.l.c., the use of programmed multiple development, and ways of improving sensitivity (*e.g.*, use of fluorescence densitometry). As an example, the determination of carbamazepine in serum is cited. (26 references.) B. H.

1D33. **Luminescence assay of drugs.** Bridges, James W. (Dept. Biochem., Univ. Surrey, Guildford, England). *Methodol. Dev. Biochem.*, 1976, **5**, 25–38.—In this review, particular reference is made to minimisation of blank values when a drug is extracted from biological samples. Both fluorescence and phosphorescence are considered, and an appraisal is made of drugs amenable to determination by phosphorescence. (53 references.) B. H.

1D34. **Affinity methods for drug assay.** Butler, Vincent P., jun. (Dept. Med., Coll. Physicians & Surgeons, Columbia Univ., New York, N.Y. 10032, U.S.A.). *Methodol. Dev. Biochem.*, 1976, **5**, 71–85.—In this review, the principles of such methods are outlined, and various drugs and toxins that have been so determined are listed. Methods for carrying out radio-immunoassays are discussed, and competitive protein-binding procedures are briefly considered. (99 references.) B. H.

1D35. **Preparation of 'polyvalent' antibodies for use in simultaneous multiple-drug radio-immunoassay.** Kaul, Balkrishena; Davidow, Bernard; and Millian, Stephen J. (Dept. Health, Public Health Lab., New York, N.Y. 10016, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (1), 14–19.—Details are given of the preparation of antisera capable of binding opiates, barbiturates, cocaine and diazepam, and their metabolites, alone and/or in combination. The application of such antisera in screening tests on human urine is reported and compared with the use of single-drug immunoassay kits. B. H.

1D36. **Polarography as applied to analysis of certain drugs and their metabolites in body fluids.** Smyth, M. R.; and Smyth, W. Franklin (Chem. Dept., Chelsea Coll., London, S.W.3, England). *Methodol. Dev. Biochem.*, 1976, **5**, 15–24.—Factors affecting the cited use of polarography are reviewed, and the need for separation of the drug or metabolite from interfering material is stressed. (51 references.) B. H.

1D37. **Assay studies on urinary methyladrenalines.** Aziz, A. A. A.; Leppard, J. P.; and Reid, Eric (Wolfson Bioanal. Centre, Univ. Surrey, Guildford, England). *Methodol. Dev. Biochem.*, 1976, **5**, 159–178.—Methods for determining these compounds are reviewed, and attempts to establish procedures based on h.p.l.c., with u.v. detection, are described. (46 references.) B. H.

1D38. **Evaluation of the Hall electrolytic conductivity detector for analysis of narcotic alkaloid and phenothiazine drugs extracted from urine.** Risk, Carol A.; and Hall, Randall C. (Dept. Entomol., Purdue Univ., West Lafayette, Ind. 47907, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (5), 156–159.—Overdose levels (1 to 4 mg dl^{-1}) of the drugs are extracted from urine (5 ml), after addition of $NaHCO_3$ - K_2CO_3 (3:2; 1 to 2 g), into benzene (8 ml). Therapeutic levels (0·1 to 0·4 mg dl^{-1}) are extracted from urine (5 ml), buffered as above, into benzene (3 × 10 ml), the combined extracts are dried over Na_2SO_4 (1 g), a portion (15 ml) is evaporated to dryness, and the residue is re-dissolved in 4% ethanol in hexane (0·5 ml). These test soln. (4 μl) are subjected to g.l.c. on a glass column (6 ft. × 2 mm) packed, preferably, with 5% of OV-101 on Gas-Chrom Q (80 to 100 mesh) and operated at 235° (narcotic alkaloids) or 260° (phenothiazine drugs) with He (60 ml min^{-1}) as carrier gas. The detector is operated in the catalytic reductive mode with a silica reaction tube packed with nickel wire in the middle or hot zone of the furnace (800°) and with a

nickel reaction tube; the conductivity solvent was aq. 10% propanol (0·5 ml min^{-1}). The lower limit of detection was 0·1 mg dl^{-1} and recoveries ranged from 38% for cocaine to 102% for methapyrilene at the 1 mg dl^{-1} level. M. J. Sl.

1D39. **Radio-immunoassay of hydromorphone in plasma.** Honigberg, I. L.; Stewart, J. T.; Brown, W. J.; Jun, H. W.; Needham, T. E.; and Vallner, J. J. (Sch. Pharm., Univ. Georgia, Athens, Ga. 30602, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (2), 70–72. —A commercial kit for determining morphine (Abuscreen No. 43021; Roche Diagnostics) has been adapted for use in determining hydromorphone (10 to 40 ng ml^{-1}) in 0·1-ml samples. Full details are given. B. H.

1D40. **Novel quinidine metabolism in a suicide case with quinidine sulphate detected by gas chromatography - mass spectrometry.** Leferink, J. G.; Maes, R. A. A.; Sunshine, Irving; and Forney, R. B., jun. (Centre Human Toxicol., State Univ., Utrecht, Netherlands). *J. Anal. Toxicol.*, 1977, **1** (2), 62–65.— Ante-mortem samples of serum and urine, and post-mortem blood, urine, brain, spleen, kidney and liver, were examined by a method involving extraction from alkaline medium with $CHCl_3$ - isopropyl alcohol (2:1). The extracts were evaporated, the residues were acidified, and the acid soln. were extracted with $CHCl_3$. These $CHCl_3$ extracts were submitted to g.l.c. at 240° on a conventional column (3% of OV-1), and the effluents were examined by m.s. (electron impact, and chemical ionisation with CH_4 as reactant gas). As well as the expected quinidine and 3-hydroxyquinidine, the lactate conjugates of both these compounds were identified. B. H.

1D41. **Trace-enrichment techniques in reversed-phase high-performance liquid chromatography.** Schauwecker, P.; Frei, R. W.; and Erni, F. (Groupe Rech. Anal. Mater., Dept. Chim., Univ. Montreal, Que., Canada). *J. Chromatogr.*, 1977, **136** (1), 63–72. —Ergot alkaloids, peptides and deproteinised urine samples were subjected to h.p.l.c. on columns (3 to 15 cm × 3 or 4 mm) slurry-packed with RP-8 (Merck, 5 μm diam.) or Nucleosil C_{18}, with acetonitrile - 0·1M-$(NH_4)_2CO_3$ as mobile phase and spectrophotometric detection. Up to 165 ml of soln. was injected and subjected to step-gradient elution (Erni *et al.*, *Ibid.*, 1976, **125**, 265). The limit of detection of dihydroergocristine mesylate in urine was 100 p.p.b.; the coeff. of variation was <2% (9 determinations). Nonapeptides were converted into derivatives with fluorescamine, which improved the detection limit by a factor of 50 without significant loss of reproducibility. M. J. Sl.

1D42. **Determination of colchicine in biological fluids.** Bourdon, R.; and Galliot, N. (Lab. Biochim.-Toxicol., Hop. Fernand-Widal, Paris, France). *Ann. Biol. Clin. (Paris)*, 1976, **34** (6), 393–401 (in French). —The method is based on spectrofluorimetry (at 620 nm, with excitation at 510 or 530 nm and correction for the reading at 565 nm) of a complex formed between colchicine, Ga and isoniazid. Procedures are described in detail for application to urine (two variants, only one of which involves initial extraction of the sample) and to plasma (with addition of phenylbutazone to displace colchicine from its protein conjugates). Possible interference from, *e.g.*, phenothiazine drugs or metabolites of colchicine is discussed. E. J. H. B.

1D43. **Evaluation of selected methods for determining cocaine and benzoylecgonine in urine.** Wallace, Jack E.; Hamilton, Horace E.; Christenson, James G.; Shimek, Eugene L., jun.; Land, Pam; and Harris, Steven C. (Dept. Pathol., Univ. Texas Health Sci. Center, San Antonio, Tex. 78284, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (1), 20–26.—The methods studied were t.l.c. (*cf. Anal. Abstr.*, 1976, **30**, 6D17), g.l.c. (*Ibid.*, 1976, **31**, 2D16), enzyme immunoassay with Syva kits, and radio-immunoassay by the method of Cleeland *et al.* [*Clin. Chem.* (*Winston-Salem, N.C.*), 1976, **22**, 712] with Roche reagents. The g.l.c. method was the most precise, and t.l.c. was the most generally applicable for determining several drugs concurrently. Enzyme immunoassay was preferred for rapidity when analysing small numbers of samples, but radio-immunoassay was the most sensitive procedure and the most efficient for assaying moderate to large numbers of samples. B. H.

1D44. **[Methods for use in] diagnosis and management of recreational mescaline self-poisoning.** Teitelbaum, Daniel T.; and Wingeleth, Dale C. (POISONLAB, Chemed Corp., Denver, Colo. 80222, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (1), 36–37.— Urine is buffered at pH 9·3 with 0·01M-carbonate, and mescaline is extracted into $CHCl_3$ - isopropyl alcohol (3:1). The extract is evaporated at room temp., and an aliquot of a soln. of the residue in methanol, together with authentic mescaline, is submitted to t.l.c. on silica gel, with ethyl acetate - methanol - aq. NH_3 (17:1:1) as mobile phase. After development for 10 cm, the chromatogram is dried and sprayed with ethanolic ninhydrin, then irradiated at 350 nm. Mescaline gives a pink spot, and its presence is confirmed by further t.l.c., with $CHCl_3$ - methanol (9:1) as mobile phase and Marquis reagent for detection (as an orange spot). Further confirmation can be obtained by g.l.c. (at 150°) of a methanolic soln. of the extract residue on a PTFE-lined aluminium column packed with Chromosorb W AW coated with 3% of SE-30, with He as carrier gas (60 ml min^{-1}) and flame ionisation detection; mescaline yields four peaks, with retention times of 2·5, 3·5, 4·5 and 7 min. B. H.

1D45. **Fluorimetric assay and pharmacokinetics of vincamine in rats.** Iven, H.; and Siegers, C.-P. (Abt. Pharmakol., Med. Hochsch., Luebeck, W. Germany). *Arzneim.-Forsch.*, 1977, **27(I)** (6a), 1248–1254 (in German).—Vincamine (**I**), which does not fluoresce, is a methyl ester. Hydrolysis for 15 min at room temp. with 0·1N-NaOH yields the corresponding acid (**II**); **II** fluoresces intensely at 372 nm, with excitation at 282 nm, and can thus serve as the basis for a sensitive method for the determination of **I** in body fluids. The main metabolite of **I** is **II**, but, by shaking the sample with heptane - isoamyl alcohol (97:3), **I** can be extracted without contamination by **II**. The procedure was applied to pharmacokinetic studies after intravenous injection of **I**. F. P.

1D46. **Gas chromatography - mass fragmentographic method for ajmaline and its chloroacetyl ester.** Clemans, St. D.; Davison, C.; Koss, R. F.; Dorrbecker, B.; O'Melia, P. E.; Ross, R. W., jun.; and Edelson, J. (Sterling-Winthrop Res. Inst., Rensselaer, New York, N.Y. 12144, U.S.A.). *Arzneim.-Forsch.*, 1977, **27 (I)** (6), 1128–1130.—The method was devised for determining ajmaline (**I**) and its 17-chloroacetate (**II**) in heparinised blood. Details are given for the extraction of **I** and **II** from blood into CH_2Cl_2 (containing oestradiol as internal

standard) and for the preparation of their trimethylsilyl derivatives, which are submitted to g.l.c. at 250° on a glass column (6 ft. × 2 mm) packed with 3% of OV-1 on Gas-Chrom Q (100 to 120 mesh) with He as carrier gas (40 ml min^{-1}). The effluent is examined by m.s., with monitoring at *m/e* values of 470·3, 474·2 and 416·2 for the derivatives of **I**, **II** and oestradiol, respectively. Calibration graphs (peak-area ratios) are rectilinear for up to ≃170 ng ml^{-1} of **I** or ≃330 ng ml^{-1} of **II** in blood. The method was applied to analysis of canine blood after an intravenous dose of 2 mg kg^{-1} or an oral dose of 5 mg kg^{-1}. G. E. F.

1D47. **Rapid micro-method for high-performance liquid-chromatographic determination of theophylline in human serum.** Cooper, M. J.; Mirkin, B. L.; and Anders, M. W. (Dept. Pharmacol., Univ. Minnesota Health Sci. Center, Minneapolis, Minn. 55455, U.S.A.). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 324–326.—The serum is deproteinised with $HClO_4$ and the supernatant soln. is neutralised with K_2CO_3; the $KClO_4$ is centrifuged off, and an aliquot of the supernatant soln. is submitted to h.p.l.c. on a column (30 cm × 4 mm) of μBondapak C_{18}, with methanol - 10mM-NaH_2PO_4 (1:4) as mobile phase (0·8 ml min^{-1}) and detection at 280 nm. The limit of detection is 1 μg ml^{-1}, and as little as 50 μl of sample can be analysed. The recovery of 1 to 25 μg ml^{-1} of added theophylline is 88 ± 5% (16 determinations). B. H.

1D48. **Determination of digitoxin in human serum.** Faber, D. B.; de Kok, A.; and Brinkman, U. A. T. (Lab. Toxicol. and Biopharm., Acad. Hosp., Free Univ., Amsterdam, Netherlands). *Methodol. Dev. Biochem.*, 1976, **5**, 247–248.—The sample is extracted with $CHCl_3$, the extract is evaporated to dryness at 40° in a stream of air, and a soln. of the residue in $CHCl_3$ is submitted to t.l.c. on Kieselgel 60, with $CHCl_3$ - methanol - acetone - H_2O (32:3:14:1) as mobile phase. The chromatogram is exposed to HCl vapour and irradiated for 12 min with a quartz - halogen lamp, then heated to remove acid and scanned fluorimetrically at 536 nm (with excitation at 365 nm). Digitoxin in amounts from 2 to 20 ng is determined by comparing the fluorescence with that of standard spots. R_F values are reported for digitoxin, digitoxigenin, digitonin, digoxin and digitoxigenin mono- and bis-digitoxosides. B. H.

1D49. **Radio-enzymic assay techniques for [antibiotic] aminoglycosides.** Forrey, Arden W.; Blair, Andrew D.; O'Neill, Michelle A.; and Cutler, Ralph E. (Dept. Med., Univ. Washington, Harborview Med. Center, Seattle, Wash. 98104, U.S.A.). *Methodol. Dev. Biochem.*, 1976, **5**, 235–240.—Procedures for the determination of therapeutic levels (≃10 mg l^{-1}) of antibiotics of the streptomycin, spectinomycin, kanamycin and gentamicin groups in plasma and urine are reviewed. The methods involve enzyme-catalysed introduction of a labelled grouping (*e.g.*, adenyl or phosphate) into the molecule and separation of the labelled product by paper chromatography before counting. B. H.

1D50. **Microbiological assay of cephradine in blood and urine.** Forster, Thomas C. (Squibb International Dev. Lab., Moreton, Merseyside, England). *Methodol. Dev. Biochem.*, 1976, **5**, 227–230.—The sample is diluted with 0·1M-phosphate buffer of pH 6·0, then assayed by an agar-plate diffusion method with *Sarcina lutea* as test organism and incubation for 16 h at 30°; the sensitivity is 0·05 μg ml^{-1} for serum and 3 μg ml^{-1} for urine. When analysing pharmaceutical preparations, *Bacillus pumilus* is used as test organism. B. H.

1D51. **Determination of cephaloridine in serum and tissue by high-performance liquid chromatography.** Wold, J. S.; and Turnipseed, S. A. (Toxicol. Div., Res. Lab., Eli Lilly and Co., Greenfield, Ind. 46140, U.S.A.). *J. Chromatogr.*, 1977, **136** (1), 170–173.—Homogenates (1:10) of rabbit renal cortex in H_2O were prepared, and an equal vol. of 6% trichloroacetic acid was added. The soln. was kept in an ice-bath for 15 min then centrifuged at 1800 r.p.m. for 10 min. The supernatant liquid was subjected to h.p.l.c. on a stainless-steel column (60 cm × 2 mm) packed with phenyl/Corasil (37 to 50 μm) with 18 to 20% methanol in aq. 0·2M-ammonium acetate as mobile phase (2 ml min^{-1}), and the effluent was monitored spectrophotometrically at 254 nm. The calibration graph for cephaloridine in serum was rectilinear from 2 to 100 μg ml^{-1}, and the recovery was 98%. The coeff. of variation (8 determinations) was 3·9% for serum (35 μg ml^{-1}) and 2·8% for tissue homogenate (1600 μg ml^{-1}). M. J. Sl.

1D52. **Plasma prednisolone concentrations: comparison of radio-immunoassay and competitive protein-binding assay.** Tembo, A. V.; Ayres, J. W.; Sakmar, E.; Hallmark, M. R.; and Wagner, J. G. (Coll. Pharm. and Upjohn Center Clin. Pharmacol., Univ. Michigan, Ann Arbor, Mich. 48109, U.S.A.). *Steroids*, 1977, **29** (5), 679–693.—A statistical study is presented of the results of assays by these techniques (each with use of an accepted procedure). It is concluded that the protein-binding method is more precise than the radio-immunoassay and is suitable for studies on prednisolone and prednisone. The steroid-binding globulin cross-reacted to a much lesser extent with the major metabolite of prednisolone than did the antiserum used in the radio-immunoassay (*cf.* Colburn and Buller, *Ibid.*, 1973, **21**, 833). B. H.

1D53. **Analysis of prednisolone in plasma by high-performance liquid chromatography.** Loo, J. C. K.; Butterfield, A. G.; Moffatt, J.; and Jordan, N. (Pharm. Chem. Div., Drug Res. Lab., Health Protect. Branch, Ottawa, Ont., Canada). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 275–280.—Plasma plus 20β-dihydrocortisone (internal standard) is extracted with ethyl ether - CH_2Cl_2 (3:2), and the organic layer is washed with 0·1M-HCl, then evaporated to dryness under N at 55°. A soln. of the residue in the mobile phase [hexane - CH_2Cl_2 - ethanol - acetic acid (319:150:30:1)] is submitted to h.p.l.c. on a column (25 cm × 3·2 mm) of LiChrosorb SI-60 (5 μm), with elution at 2 ml min^{-1} and detection at 254 nm. Prednisolone (eluted in ≃6 min) is well separated from prednisone, hydrocortisone, dexamethasone, the internal standard and 20β-dihydroprednisolone. The calibration graph (peak-height ratios) is rectilinear for 25 to 200 ng ml^{-1} in the sample. B. H.

1D54. **High-performance liquid-chromatographic analysis for synthetic corticosteroids in plasma.** Loo, J. C. K.; and Jordan, N. (Pharm. Chem. Div., Drug Res. Lab., Health Protect. Branch, Ottawa, Ont., Canada). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 314–316.—The method of Loo *et al.* for prednisolone (**I**) (see preceding abstract) has been

adapted for the determination of (*a*) dexamethasone (**II**), and (*b*) the simultaneous determination of **I** and prednisone. The internal standard is β-methasone in (*a*) and **II** in (*b*), and the mobile phase (2·5 ml min^{-1}) is anhyd. acetic acid - ethanol - CH_2Cl_2 - hexane [1 : 20 : 150 : 329 in (*a*); and 2 : 35 : 300 : 663 in (*b*)]. The limits of determination (ng ml^{-1}) are 25 for **II** and prednisone and 100 for **I**.
B. H.

1D55. **Assay of methaqualone in blood.** Brown, Stanley S. (Div. Clin. Chem., Clin. Res. Centre, Harrow, Middx., England). *Methodol. Dev. Biochem.*, 1976, **5**, 179–184.—Methods for this assay are briefly reviewed. The procedure recommended involves extraction of the drug from serum or plasma into, *e.g.*, hexane, re-extraction into acid, and further extraction into an organic solvent (to separate the drug from phenolic metabolites) for determination by u.v. spectrophotometry, fluorimetry (after reduction to a dihydro-derivative) or g.l.c. (15 references.)
B. H.

1D56. **Determination of phenobarbitone and phenytoin in serum by a mechanised enzyme immunoassay in comparison with a gas - liquid chromatographic method.** Oellerich, M.; Kuelpmann, W. R.; Haeckel, R.; and Heyer, R. (Inst. f. Klin. Chem., Med. Hochsch., Hannover, W. Germany). *J. Clin. Chem. Clin. Biochem.*, 1977, **15** (6), 353–358.—The assay of phenytoin (**I**) and phenobarbitone (**II**) with EMIT kits (Syva Corp., Palo Alto, Calif., U.S.A.) has been automated by using an Eppendorf analyser 5010. Serum (10 μl) was incubated for 15 min at 15° with 500 μl of reagent A (antibody - glucose 6-phosphate - NAD^+, diluted 1 : 13·5 with Tris-HCl buffer of pH 7·9). Reaction was then started with 100 μl of reagent B, containing glucose-6-phosphate dehydrogenase coupled to **I** or **II** (reagent B was diluted 1 : 2·5 with Tris-HCl buffer of pH 7·9); the absorbance at 334 nm was monitored. Calibration graphs were rectilinear up to 80 mg l^{-1} of **II** and up to 30 mg l^{-1} of **I** in serum. More concentrated samples were diluted with bovine albumin soln. Within-series coeff. of variation were 6 to 13%, and recovery of 10 to 50 mg l^{-1} of drug added to sera was 93 to 99% for **II** and 93 to 101% for **I**. The results agreed well with those obtained by g.l.c. on SP 2250 DA on Chromosorb W HP. Of 27 drugs tested, methylphenobarbitone cyclobarbitone, cyclopentobarbitone and amylobarbitone showed cross-reactivity with the assay of **II**, and, of 19 tested for cross-reactivity with the assay of **I**, only methoin and 5-(4-hydroxyphenyl)-5-phenylhydantoin showed reaction.
T. H. N.

1D57. **Phenytoin. Comparison study of the enzyme immunoassay and a chemical method.** Vasiliades, John; Shettlesworth, David; and Owen, K. (Dept. Pathol., Univ. Alabama, Birmingham, Ala. 35233, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (2), 73–74.—The semi-automated EMIT system (Syva Corp., Palo Alto, Calif., U.S.A.) has been compared with the spectrophotometric method of Wallace and Hamilton (*J. Pharm. Sci.*, 1974, **63**, 1795) for this assay. The metabolite 5-(4-hydroxyphenyl)-5-phenylhydantoin caused a positive error (≃12%) in the EMIT method but not in the chemical method. Drugs other than phenytoin likely to be present interfered in neither method. There was good correlation between results by the two procedures, and it is concluded that they can be used interchangeably in routine assays.
B. H.

1D58. **Toxicological determination of benzodiazepines in biological fluids and tissues by flame ionisation [detection after] gas chromatography.** Basalt, Randall C.; Stewart, Constance B.; and Franch, Serafino J. (Dept. Lab. Med., Univ. Connecticut Health Center, Farmington, Conn. 06032, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (1), 10–13.—The sample fluid or aq. homogenate of tissue, plus prazepam as internal standard, is made alkaline with conc. aq. NH_3 and extracted with 1-chlorobutane. The extract is shaken with M-HCl, then the HCl layer is made alkaline with aq. NH_3 and shaken with $CHCl_3$. An aliquot of the $CHCl_3$ soln. is submitted to g.l.c. at 260° on a glass column (6 ft. × 2 mm) packed with 2% of OV-17 on Chromosorb G-HP, with N as carrier gas (33 ml min^{-1}) and flame ionisation detection. Retention times (2·8 to 22 min) and detection limits (0·2 μg ml^{-1}) are reported for medazepam, oxazepam, diazepam, 'dealkylflurazepam' (a metabolite), desmethyldiazepam, chlordiazepoxide, prazepam, flunitrazepam, flurazepam, nitrazepam and clonazepam. R_F values for these compounds in t.l.c. on Silica gel G, with acetone - benzene - $CHCl_3$ (5 : 8 : 8) as mobile phase, are also reported for use in confirming the identity of compounds separated by g.l.c.
B. H.

1D59. **Determination of two benzodiazepine anticonvulsants in plasma of rhesus monkeys when given intravenously, singly or together or in combination with a benzhydrylpiperazine anticonvulsant.** Clifford, J. M. (G. D. Searle and Co., High Wycombe, Bucks., England). *Methodol. Dev. Biochem.*, 1976, **5**, 203–209.—A method is described for analysing samples containing diazepam and/or clonazepam (and their metabolites) in conjunction with SC 13504 [1-benzhydryl-4-(6-methyl-2-pyridylmethyleneamino)piperazine]. It is based on selective extraction of the compounds with light petroleum and ethyl ether (*cf.* Barrett *et al.*, *J. Pharm. Pharmacol.*, 1974, **26**, 9) and subsequent differential pulse polarography [Smyth *et al.*, *Analyst* (*London*), 1976, **101**, 469]. A scheme based on that of Naestoft and Larsen (*Anal. Abstr.*, 1975, **28**, 2D35) might be suitable for determining the metabolites.
B. H.

1D60. **Determination of primidone [in blood plasma] by electron-capture gas chromatography.** Wallace, Jack E.; Hamilton, Horace E.; Shimek, Eugene L., jun.; and Schwertner, Harvey A. (Dept. Pathol., Univ. Texas Health Sci. Center, San Antonio, Tex. 78284, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 903–906.—The method is based on derivatisation of the compound with pentafluorobenzoyl chloride after extraction from the sample into 20% ethyl acetate soln. in toluene or benzene, with $(NH_4)_2SO_4$ being used as the salting-out agent. The derivative is analysed on a glass column (1 m × 4 mm) packed with 3% of OV-1 on Gas-Chrom Q (100 to 120 mesh) and operated at 220°, with Ar - CH_4 (19 : 1) as carrier gas (60 ml min^{-1}) and ^{63}Ni electron-capture detection. Recoveries are >92%, and the coeff. of variation are usually <1% (from quadruplicate analyses of 12 samples containing 0 to 20 μg ml^{-1} of primidone).
G. P. C.

1D61. **Determination of acetaminophen [paracetamol] in serum.** Walberg, Clifford B. (Toxicol. Lab., Los Angeles County–Univ. S. California Med. Center, Los Angeles, Calif. 90033, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (2), 79–80.—The procedure is a modification of the colorimetric method of Glynn and Kendal (*Lancet*, 1975, **I**, 1147). Changes in the concn. and amounts of reagents have been made

whereby the absorbance at 430 nm has been increased from 0·16 (with 12·5 ml of final soln.) to 0·44 (4·5 ml of soln.) for 10 mg dl^{-1} of paracetamol. The calibration graph is rectilinear for up to 60 mg dl^{-1}, and this range can be extended to 100 mg dl^{-1} by diluting the final soln. Calibration is effected with standard soln. in serum. The method is not recommended for absolute determinations, but is useful for identification of paracetamol and for establishing whether or not an overdose has been ingested. B. H.

1D62. **Isolation and identification of two hydroxylated metabolites of phenazopyridine in rat urine.** Pitre, D.; and Maffei-Facino, R. (Res. Lab., Bracco Industria Chimica S.p.A., Milan, Italy). *Farmaco, Ed. Sci.*, 1977, **32** (6), 453–460.—The metabolites, 2,6-diamino-3-(2-hydroxyphenylazo)pyridine and its 4-hydroxyphenylazo-analogue, were isolated from the sample by t.l.c. on Kieselgel F_{254} after extraction into CH_2Cl_2, evaporation of the extract at 30° and dissolution of the residue in methanol. R_F values are given for these compounds, the unchanged analgesic, 2,3,6-triaminopyridine and aniline in three mobile phases. The zones were detected by spraying with, *e.g.*, $FeCl_3$ soln. or by exposure to u.v. radiation or HCl fumes. The hydroxylated compounds were identified by i.r. spectrophotometry (KBr-disc technique), 60-MHz n.m.r. and 70-eV m.s. B. H.

1D63. **Quantitative gas-chromatographic determination of nefopam in plasma.** Ehrsson, Hans; and Eksborg, Staffan (Karolinska Pharmacy, Stockholm, Sweden). *J. Chromatogr.*, 1977, **136** (1), 154–158.—Plasma (5 ml) was mixed with 0·2 ml of a 1·31-p.p.m. aq. soln. of the hydrochloride of the *N*-isopropyl analogue of nefopam, as internal standard, and M-NaOH (0·1 ml) was added. The soln. was shaken with toluene (5 ml) for 30 min and the separated organic layer was extracted with phosphate buffer soln. of pH 7·3 (5 ml) for 10 min, and with 0·1M-H_2SO_4 (0·1 ml) for 10 min. The acid phase was flushed with N to remove toluene, then mixed with M-NaOH and CH_2Cl_2 (25 μl of each) and shaken for 5 min. The organic phase was subjected to g.l.c. on a glass column (90 cm × 2 mm) packed with 3% of OV-225 on Gas-Chrom Q (100 to 120 mesh) and operated at 170°, with N as carrier gas (25 ml min^{-1}) and a flame ionisation detector. The recovery of nefopam (10 and 100 ng ml^{-1}) was 90%, and the calibration graph prepared for 5 to 50 ng ml^{-1} of nefopam in plasma was rectilinear. The coeff. of variation (5 determinations) at levels of 10 and 50 ng ml^{-1} were 2·2 and 2·7%, respectively. M. J. St.

1D64. **Comparison of methods used in detection of methadone and its primary metabolite [2-ethylidene-1,5-dimethyl-3,3-diphenylpyrrolidine].** Jain, Naresh C.; Leung, Wai J.; Sneath, Thomas C.; Budd, Robert D.; and Chinn, Dennis (Rancho Los Amigos Hosp., Downey, Calif. 90242, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (1), 6–9.—Urine was analysed for these two compounds by enzyme immunoassay, haemagglutination inhibition, radio-immunoassay, t.l.c. and g.l.c.; the results are discussed. If detection of both compounds in a large number of samples is required, or if many other drugs are to be tested for, t.l.c. is preferred. If only methadone is to be detected in large numbers of samples, radio-immunoassay (Roche Abuscreen kit) is recommended. For small numbers of samples, and/or when accurate determination is required, g.l.c. should be used. B. H.

1D65. **Gas-chromatographic method for determination of methadone and its metabolites in biological fluids and tissues.** Thompson, Bernard C.; and Caplan, Yale H. (Office Chief Med. Examiner, State of Maryland, Baltimore, Md. 21201, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (2), 66–69.—The procedure involves extraction (at pH 6·8) of blood, urine, bile, stomach contents or tissue homogenate [each with proadifen hydrochloride (**I**) added as internal standard] with 1-chlorobutane. The extract is washed with N-H_2SO_4, and the acid soln. is neutralised with 4N-NaOH; the pH is adjusted to 9·0 (carbonate buffer soln.), and the mixture is extracted with $CHCl_3$ containing docosane (as second internal standard). This extract is submitted to g.l.c. at 210° on a glass column (4 ft. × 2 mm) packed with 3% of OV-17 on Gas-Chrom Q (100 to 120 mesh), with N (30 ml min^{-1}) as carrier gas and flame ionisation detection. The peak heights are measured for **I**, methadone (**II**), and its metabolites (2-ethylidene-1,5-dimethyl-3,3-diphenylpyrrolidine and 2-ethyl-5-methyl-3,3-diphenylpyrroline) relative to docosane. For **II**, the detection limit is 0·3 ng (equiv. to 20 ng ml^{-1} in the sample). Recovery of **II** ranged from 64% (from brain) to ≃87% (from urine) for 0·08 to 8 μg ml^{-1} added. There is no interference from 10 common basic drugs that would be co-extracted with **II**, and the procedure could be used in determining these drugs also. B. H.

1D66. **Accumulation of acetylmethadol [methadyl acetate] and active metabolites in plasma following chronic administration [of methadone].** Henderson, Gary L.; Weinberg, J. Arthur; Hargreaves, William A.; Lau, Derick H. M.; Tyler, Joan; and Baker, Barbara (Dept. Pharmacol., Sch. Med., Univ. California, Davis, Calif. 95616, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (1), 1–5.—The methods used in this study were as follows. Plasma (at pH 9·5) is extracted with 1-chlorobutane, the extract is evaporated under N, and an aliquot of a soln. of the residue in acetone is submitted to g.l.c. at 190° on a glass column (3 ft. × 0·125 in.; 3% of OV-25 on Gas-Chrom Q), with N as carrier gas (40 ml min^{-1}) and flame ionisation detection. The retention times are 19·9 min for methadone and 23·2 min for methadyl acetate (**I**), and the detection limit is 35 ng ml^{-1} for either compound; the calibration graph is rectilinear for up to 200 ng ml^{-1} of **I**. To determine the noracetyl and dinoracetyl metabolites of **I**, another extract of plasma is evaporated to dryness, and the residue is treated for 1·5 h at 70° with trichloroacetyl chloride in toluene. This mixture is evaporated to dryness, and a soln. of the residue in hexane is submitted to g.l.c. at 240° on a similar column, with 3% of OV-17 as stationary phase, ^{63}Ni electron-capture detection and Ar - CH_4 as carrier gas (40 ml min^{-1}). The retention times are 11·2 and 8·5 min for the noracetyl and dinoracetyl metabolites, respectively. The detection limit for each compound is 10 ng ml^{-1} and each calibration graph is rectilinear for up to 200 ng ml^{-1}. B. H.

1D67. **Separation of radio-labelled acetylmethadol [methadyl acetate] and metabolites by thin-layer chromatography.** Flor, S. Campos; and Inturrisi, C. E. (Dept. Pharmacol., Cornell Univ. Med. Coll., New York, N.Y. 10021, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (2), 75–76.—[^{3}H]Methadyl acetate and seven of its known or possible metabolites have been separated on Kontes pre-coated silica gel plates having a 'pre-adsorbent' layer of diatomaceous earth, to which the sample, *e.g.*, urine or baboon bile, is applied directly (after addition of the

authentic compounds as carriers). The plates are developed twice in the same direction with hexane - ethyl ether - acetone - diethylamine (180 : 75 : 40 : 4) and dried in air, then the spots are located with Dragendorff reagent and scraped off for scintillation counting. R_F values are reported. B. H.

1D68. [**Methods used in**] **a fatal case involving levorphanol.** Turner, J. E.; and Richards, Robert G. (Orange County Sherriff-Coroner, Santa Ana, Calif. 92702, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (2), 103–104.—Results are reported for levorphanol (**I**) in blood, bile, urine, stomach contents and organs. **I** was detected by making the sample alkaline with NaOH, extracting with $CHCl_3$, shaking the H_2O-washed extract with 0·1M-H_2SO_4 and recording the u.v. spectrum of the acid soln.; **I** shows max. absorption at 279 nm, with an inflection at 285 nm. Confirmation is by t.l.c. on silica gel (with fluorescent indicator), with methanol containing 1·5% of satd. NH_3 soln. as mobile phase; the R_F value for **I** is 0·32, and spraying with acidified iodoplatinate reagent gives a deep-purple spot. Determination of **I** is by g.l.c. at 250° on a conventional column (3% of OV-10 on Gas-Chrom Q), with He as carrier gas and codeine as internal standard. The preparation of sample extracts for g.l.c. is described. B. H.

1D69. **Quantitative determination of fenclorac [α,3-dichloro-4-cyclohexylphenylacetic acid] in serum.** Lee, J. K.; Smyth, R. D.; Polk, A.; Herczeg, T.; Tsuei, C. T.; and Reavey-Cantwell, N. H. (William H. Rorer Inc., Fort Washington, Pa. 19034, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 832–834.—The method of Cummins and Perry (*Anal. Abstr.*, 1970, **19**, 1701) for the determination of pemoline has been modified. *Procedure*—Mix the sample with methanol, centrifuge, and mix the supernatant liquid with H_2O, 8N-HCl and $CHCl_3$; centrifuge and extract the separated lower phase with N-NaOH. Heat the combined alkaline soln. in boiling water for 15 min to hydrolyse the salt of fenclorac (**I**) to one of (3-chloro-4-cyclohexylphenyl)glycollic acid (**II**), cool to room temp., wash the soln. with hexane, and reject the washings. Acidify with 8N-HCl, add 0·2M-$(NH_4)_2SO_4.Ce(SO_4)_2$, and, after 30 min in the dark, add 10N-NaOH, mix, extract the oxidation product (3-chloro-4-cyclohexylbenzaldehyde) into hexane and measure the absorbance at 252 nm. The over-all recovery in the extraction procedure is ≃75%. **II**, a minor metabolite of **I**, interferes. The coeff. of variation (12 to 14 determinations at each of 5 concn. from 2 to 30 μg ml^{-1}) was 5%. A. R. R.

1D70. **Problems in analysis of *N*-oxygenated products of phenothiazines.** Cowan, David A. (Dept. Pharm., Chelsea Coll., London, S.W.3, England). *Methodol. Dev. Biochem.*, 1976, **5**, 193–201.—The metabolism of phenothiazines is discussed, and a procedure is described for determining, *e.g.*, chlorpromazine and its *N*-oxide, *S*-oxide and *NS*-dioxide. The method involves extraction of the sample (at pH 4) with benzene - dichloroethane (19 : 1) to separate the drug and its *N*-oxide (in the organic layer) from the *S*-oxides. Further extractions are used to separate the drug from its *N*-oxide for assay by g.c., and the various oxides are determined by polarography (*cf.* Beckett *et al.*, *Anal. Abstr.*, 1975, **28**, 1D39). B. H.

1D71. **Assay of chlorpromazine and some of its metabolites in biological fluids.** Curry, S. H. (Dept. Pharmacol. and Therap., London Hosp. Med. Coll., London, E.1, England). *Methodol. Dev. Biochem.*, 1976, **5**, 185–192.—A review is presented, with 30 references. The preferred procedure involves extraction of citrated or oxalated plasma (made ≃0·3M in NaOH) with 1·5% amyl alcohol soln. in heptane and back-extraction of the compounds (unchanged chlorpromazine and its demethylated and *S*-oxidised analogues) into 0·05M-HCl. The acid soln. is made alkaline with NH_3 soln., and the compounds are extracted into 15% amyl alcohol soln. in toluene; this extract is examined by g.l.c. on a conventional column (*e.g.*, 3% of OV-17). Procedures are also described for determining further metabolites in plasma and non-conjugated metabolites in urine. B. H.

1D72. **Solvent extraction as applied to fluphenazine and its metabolites.** Whelpton, R.; and Curry, S. H. (Dept. Pharmacol. and Therap., London Hosp. Med. Coll., London, E.1, England). *Methodol. Dev. Biochem.*, 1976, **5**, 115–120.—Details are given of a scheme for extracting fluphenazine into heptane and two of its metabolites (the 7-hydroxy-derivative and the *S*-oxide) into toluene; the compounds can be selectively extracted at different pH values. Extraction from urine, plasma and aq. soln. is discussed. B. H.

1D73. **Thin-layer chromatographic screening and confirmation of phencyclidine in urine.** Jain, Naresh C.; Budd, Robert D.; Leung, Wai J.; and Sneath, Thomas C. (Rancho Los Amigos Hosp., Downey, Calif. 90242, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (2), 77–78.—Urine plus Tris buffer of pH 9 is shaken with $CHCl_3$ - isopropyl alcohol (19 : 1), and the extract is evaporated to dryness under N at 70° after adding methanolic 1% HCl. A soln. of the residue in methanol is halved, the portions being applied to Silica gel G layers for separate development with ethyl acetate - methanol - diethylamine (225 : 25 : 4) and CH_2Cl_2 - butanol - conc. NH_3 soln. (425 : 75 : 1). The dried chromatograms are sprayed with iodoplatinate reagent; phencyclidine gives purple - grey spots (R_F 0·58 in the first system and 0·38 in the second). R_F values for 23 other drugs and a methadone metabolite are reported; none interferes. As little as 0·2 μg ml^{-1} of phencyclidine can be detected, and 150 to 200 samples can be analysed in ≃8 h. B. H.

1D74. **Use of high-performance liquid chromatography for sample clean-up in mass-fragmentographic assays.** de Ridder, Johannes J.; Koppens, Paul C. J. M.; and van Hal, Henk J. M. (Organon International B.V., Sci. Dev. Group, Oss, Netherlands). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 281–287.—The method was devised for use in determining the antidepressant mianserin hydrochloride (**I**). Plasma plus ^{2}H-labelled **I** is extracted with hexane, the extract is evaporated to dryness at 45° under N, and the residue is dissolved in the mobile phase [hexane - isopropyl alcohol (4 : 1) plus 4% of ethanol and 0·1% of conc. NH_3 soln. and containing amitriptyline as marker] for h.p.l.c. on a column (30 cm × 4 mm) of μPorasil; elution is at ≃2 ml min^{-1} and detection at 280 nm. The eluate is collected from 5 to 9 min after injection, and this fraction (containing **I** and amitriptyline) is evaporated to dryness, the residue being dissolved in methanol for g.l.c. - m.s. A glass column (4 m × 2 mm) packed with 1% of JXR on Gas-Chrom Q is used, and the peaks at *m/e* 264 and 266 are monitored. Recovery of ng amounts of **I** is ≃100%, but for μg amounts the recovery is poorer. Sixty samples can

be analysed in one day. The amitriptyline can be omitted once the retention time of **I** has been established. B. H.

1D75. **Mass-fragmentographic assay of nanogram amounts of the antidepressant drug mianserin hydrochloride in human plasma.** de Ridder, Johannes J.; Koppens, Paul C. J. M.; and van Hal, Henk J. M. (Organon International B.V., Sci. Dev. Group, Oss, Netherlands). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 289–297.—The drug is determined by g.l.c. - m.s. after clean-up of the sample extract by h.p.l.c. as described elsewhere (see preceding abstract). The g.l.c. column is operated at 260°, with He (30 ml min^{-1}) as carrier gas, and is connected to the 70-eV mass spectrometer by a Watson - Biemann separator. The specificity of the method and the way of establishing the calibration graph are discussed. The limit of determination is 1 ng ml^{-1} (for a 1- or 2-ml sample), but can be improved to 0·25 ng ml^{-1} by using a 4-ml sample. B. H.

1D76. **2-Dimethylaminoethanol (deanol) in body fluids.** Ceder, Gunnel; and Schuberth, Jan (Psychiat. Res. Centre, Univ. Uppsala, Ulleraker Hosp., Uppsala, Sweden). *J. Pharm. Pharmacol.*, 1977, **29** (6), 373–374.—Two methods are described for determining deanol (**I**) in the plasma and cerebrospinal fluid of rabbits and humans. *Procedures*: (*i*)—Deproteinise the sample with 20% trichloroacetic acid, and extract the supernatant liquid with ethyl ether. Evaporate the combined extracts to dryness under N, shake the residue for 10 min with methanol, centrifuge, and evaporate the supernatant liquid to dryness under N. Shake the residue for 15 min with $CHCl_3$ - 5M-NaOH (5 : 1), centrifuge, and mix a portion of the $CHCl_3$ phase with propionyl chloride. After 10 min at 22°, evaporate to dryness under N, dissolve the residue in 5 to 250 μM-**I** butanoate (**II**) soln. in $CHCl_3$ and submit this soln. to g.l.c. at 170° on a glass column (1 m × 2 mm) containing Pennwalt 223 amine packing (80 to 100 mesh), with N as carrier gas (30 ml min^{-1}) and flame ionisation detection. Compare the peak heights of **I** propionate and **II**. (*ii*)—Add $[^2H_4]$**I** as internal standard to the sample, and proceed as in (*i*), but dissolve the final residue in $CHCl_3$, and examine the g.l.c. effluent by m.s. (use He as carrier gas). The sensitivity is ≃0·05 μmol of **I**. A. R. R.

1D77. **Quantitative analysis of terbutaline in serum and urine at therapeutic levels using gas chromatography - mass spectrometry.** Leferink, J. G.; Wagemaker-Engels, I.; Maes, R. A. A.; Lamont, H.; Pauwels, R.; and Van der Straeten, M. (Centre for Human Toxicol., State Univ., Utrecht, Netherlands). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 299–305.—Serum plus $[^2H_6]$terbutaline is extracted (at pH 7·2 to 7·5) with a soln. of bis-(2-ethylhexyl) phosphate in ethyl acetate, then the extract is evaporated to dryness at 55° under N, the residue is treated with trifluorobis(trimethylsilyl)acetamide reagent at 80°, and an aliquot of the mixture is submitted to g.l.c. at 165° on a glass column (1·2 m × 3 mm) packed with 3% of OV-1 on Gas-Chrom Q (80 to 100 mesh). The effluent is examined by electron-impact m.s. (with He as carrier gas) or (preferably) by chemical-ionisation m.s. (with CH_4 as carrier and reagent gas); the choice of *m/e* values used (426, 432, 442 and 448) is discussed. The limit of detection is 1 ng ml^{-1} of terbutaline in the sample, and there is no interference from other β-receptor stimulants (*e.g.*, salbutamol, orciprenaline isoetharine or fenoterol). For urine, the extraction procedure is slightly modified. B. H.

1D78. **Rapid determination of procainamide and its *N*-acetyl derivative in human plasma by high-pressure liquid chromatography.** Weddle, Orville H.; and Mason, William D. (Sch. Pharm., Univ. Missouri–Kansas City, Kansas City, Mo. 64108, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 874–875.—Mix the sample with 5N-NaOH and butanol - $CHCl_3$ - hexane (1 : 1 : 3), centrifuge, and submit a portion of the supernatant liquid to h.p.l.c. on a stainless-steel column (30 cm × 4 mm) packed with μBondapak CN and operated with weakly ammoniacal isopropyl alcohol - hexane - methanol (1 : 1 : 3) as mobile phase (5 ml min^{-1}) and detection at 280 nm. Measure the peak heights. Mean recoveries (12 determinations of each substance at 4 concn. from 4 to 10 μg ml^{-1}) ranged from 98·3 to 101·3%, and the coeff. of variation were <3%. A duplicate determination takes ≃16 min. Quinidine, primidone, phenobarbitone and phenytoin and their metabolites, and caffeine, theobromine, theophylline, 3-methylxanthine and propranolol, did not interfere. A. R. R.

1D79. **Quantitative thin-layer chromatographic method for determination of procainamide and its major metabolite in plasma.** Wesley-Hadzija, B.; and Mattocks, A. M. (Sch. Pharm., Univ. N. Carolina, Chapel Hill, N.C. 27514, U.S.A.). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 307–313.—Plasma is made alkaline with NaOH soln. and extracted with CH_2Cl_2. The extract is evaporated to dryness at 40° under N, and a soln. of the residue in ethyl acetate is submitted to t.l.c. on Silica gel 60, together with 'spiked' samples and soln. of authentic procainamide and the metabolite (the *N*-acetyl derivative); the mobile phase is benzene - 28% NH_3 soln. - dioxan (2 : 3 : 16), and development is for 15 cm. Determination is by direct scanning of the chromatogram at 275 nm. The calibration graph is rectilinear for 10 to 200 ng of either compound on the chromatogram. B. H.

1D80. **Plasma levels of diftalone in eight animal species evaluated by thin-layer chromatography and spectrophotometric assay.** Tenconi, L. T.; Beretta, E.; Cristina, T.; and Morrone, A. (Res. Lab., Gruppo Lepetit S.p.A., Milan, Italy). *Farmaco, Ed. Prat.*, 1977, **32** (6), 284–290.—Diftalone in plasma from humans, mice, rats, hamsters, guinea-pigs, dogs, pigs and monkeys was determined after extraction into CH_2Cl_2, t.l.c. on Silica gel F_{254} (with $CHCl_3$ as mobile phase) and extraction from the layer with methanol for spectrophotometry at 232 nm. For plasma levels between 3·5 and 16 μg ml^{-1}, recoveries ranged from 87·6 to 98·9%. At levels <2 μg ml^{-1}, the u.v. absorption is within the variability of the blank, but semi-quantitative determination (by visual comparison with standards under u.v. radiation) is possible down to 0·5 μg ml^{-1}. E. C. A.

1D81. **[Determination of] niflumic acid in plasma and urine.** Cowen, T.; and Salmon, J. R. (Squibb International Dev. Lab., Moreton, Merseyside, England). *Methodol. Dev. Biochem.*, 1976, **5**, 211–216.—Plasma or urine is buffered at pH 4·0, then saturated with KCl and extracted with ethyl ether. A portion of the extract is evaporated to dryness, and the residue is dissolved in acetonitrile - 17mM-phosphate buffer soln. of pH 7 (2 : 3) (solvent *A*) for

h.p.l.c. on a column of C_{18}-treated Partisil 10, with solvent *A* as mobile phase (1 ml min^{-1}) and measurement at 286 nm. Determination is by comparison of the peak height with that for a standard soln. Niflumic acid is eluted in $\simeq$4 min and is separated from its metabolites. The range of the method is 0 to 30 μg ml^{-1}, and 60 samples can be analysed in 1 day. B. H.

1D82. **Determination of triprolidine in human plasma by quantitative thin-layer chromatography.** DeAngelis, R. L.; Kearney, M. F.; and Welch, R. M. (Dept. Med. Biochem., Wellcome Res. Lab., Durham, N.C. 27709, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 841–843.—Shake the sample (containing >0·8 ng of triprolidine) with dichloroethane for 15 min, centrifuge at 1000 *g*, and evaporate the separated organic phase to dryness at 45° in a stream of N. Dissolve the residue in $CHCl_3$, and submit a portion of the soln. to t.l.c. on a 0·25-mm layer of silica gel, with methanol - aq. NH_3 - $CHCl_3$ (10 : 1 : 89) as the mobile phase. Spray the dried chromatogram with 2M-NH_4HSO_4, dry in air for 1 h, and use a spectrodensitometer in the reflectance mode to measure the integrated fluorescence of the spot at wavelengths longer than 405 nm, with excitation at 300 nm. A. R. R.

1D83. **Simplified gas-chromatographic method for determination of chlorpheniramine in serum.** Barnhart, James W.; and Johnson, James D. (Health and Consumer Products Dept., Dow Chemical Co., Indianapolis, Ind. 46268, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1085–1086.—The method was essentially the same as that of Bruce *et al.* (*Anal. Abstr.*, 1969, **17**, 2285) except that acetylation, instead of partition chromatography, was used to remove interfering metabolites. For the determination of 10 ng ml^{-1} of chlorpheniramine in plasma, the coeff. of variation was 5·2% (10 replicate determinations), and the sensitivity was 0·5 to 1·0 ng ml^{-1}. G. P. C.

1D84. **Determination of Irgasan DP300 [triclosan] in biological fluids.** Hoar, D. R.; and Sissons, D. J. (Unilever Research, Colworth House, Sharnbrook, Bedford, England). *Methodol. Dev. Biochem.*, 1976, **5**, 221–226.—The method described was devised for determining this antiseptic in human or rat blood, but has also been applied to urine, fish tissue and fish bile. The edta-treated sample is adjusted to pH 2·2 (phosphate - citrate buffer soln.), and the free drug is extracted into ethyl ether. To determine the total drug, the sample is warmed with H_2SO_4 to hydrolyse conjugates, then the liberated drug is extracted into hexane - acetone (9 : 1); this extract is concentrated and shaken with phosphate - citrate buffer soln. of pH 6·0. The hexane layer, or the ether extract, is subjected to g.l.c. at 215° on a glass column (2·1 m × 4 mm) packed with 2·5% each of SE-30 and QF-1 and 0·02% of Epikote resin on Aeropak 30 (80 to 100 mesh), with ^{63}Ni electron-capture detection and N (60 ml min^{-1}) as carrier gas. The practical sensitivity for blood is 10 μg l^{-1}. B. H.

1D85. **Liquid chromatography in pharmaceutical analysis. VIII. Determination of isoniazid and its acetyl derivative in plasma and urine samples.** Saxena, S. J.; Stewart, J. T.; Honigberg, I. L.; Washington, J. G.; and Keene, G. R. (Dept. Med. Chem., Sch. Pharm., Univ. Georgia, Athens, Ga. 30602, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 813–816.—Mix the sample with 1-benzoyl-2-isonicotinoylhydrazine soln., dilute with H_2O, add 0·1M-NaOH and $(NH_4)_2SO_4$, and shake with H_2O-saturated butanol - $CHCl_3$ (3 : 7). Extract 30 ml of the separated lower layer with 0·1N-H_2SO_4, and submit an aliquot of the acid layer to h.p.l.c. on a μBondapak C_{18} column operated at room temp., with 0·01M-Na dioctyl sulphosuccinate in methanol - H_2O (3 : 2) at pH 2·5 as mobile phase (2 ml min^{-1}) and detection at 266 nm. Mean recoveries from plasma containing 20 μg ml^{-1} of isoniazid (**I**) and 26·6 μg ml^{-1} of the acetyl derivative (**II**) were 94·8% and 95·5%, respectively; corresponding values for urine containing 80 μg ml^{-1} of **I** and 240 μg ml^{-1} of **II** were 92·6 and 101·6%. The lower limits of detection were 200 ng of **I** and 50 ng of **II**. (For Part VII see *Anal. Abstr.*, 1977, **33**, 6D31.) A. R. R.

1D86. **Determination of ethambutol in plasma and urine by chemical-ionisation gas chromatography/mass spectrometry using a deuterated internal standard.** Blair, Andrew D.; Forrey, Arden W.; Christopher, T. Graham; Maxwell, Barbara; and Cutler, Ralph E. (Dept. Med., Univ. Washington, Harborview Med. Center, Seattle, Wash. 98104, U.S.A.). *Methodol. Dev. Biochem.*, 1976, **5**, 231–234.—The sample plus [$^2H_4$]ethambutol (prep. described) is made 2M in KOH and shaken with $CHCl_3$; the aq. layer is removed, and the $CHCl_3$ layer is treated with HCl (to prevent loss of the drug) and evaporated under N at room temp. The residue is treated with trimethylsilylimidazole - pyridine, and the mixture is submitted to g.l.c. at 140° on a conventional column (OV-17 or OV-225), the effluent being monitored by m.s. (with CH_4 as reagent gas) at *m/e* 349 and 353 (for the derivatives of ethambutol and the labelled analogue). The peaks at *m/e* 333 and 337 (M — CH_3) can also be monitored. The upper and lower limits of the method are 20 μg ml^{-1} and $\simeq$10 ng ml^{-1}, respectively. B. H.

1D87. **Quantitation of the antimalarial mefloquine in blood, plasma and urine using high-pressure liquid chromatography.** Grindel, J. M.; Tilton, P. F.; and Shaffer, R. D. (Dept. Pharmacol., Div. Med. Chem., Walter Reed Army Inst. Res., Washington, D.C. 20012, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 834–837.—To determine mefloquine (**I**) in whole blood or plasma, mix the sample with 0·065M-phosphate buffer soln. of pH 7·4 and the internal standard {4-[3-(t-butylamino)-1-hydroxypropyl]-2,8-bis(trifluoromethyl)quinoline phosphate (**II**) soln. in dioxan - methanol (7 : 3)}, and extract with ethyl acetate. Evaporate the extract to dryness at 40° in a stream of N, dissolve the residue in 0·5% acetic acid soln. in isopropyl ether - dioxan (3 : 2), and submit an aliquot to h.p.l.c. on a μBondapak CN column (30 cm × 4 mm) with the acetic acid soln. as mobile phase (2 ml min^{-1}) and detection at 280 nm. Compare the peak areas of **I** and **II**. To determine **I** in urine, proceed similarly, but use a μBondapak C_{18} column, with methanol - 0·1M-Na_2HPO_4 (3 : 2) as mobile phase. The mean recoveries and coeff. of variation (4 determinations at each of 4 concn. from 0·05 to 5 μg ml^{-1}) were—from blood, 102·9 ± 6·7%; from plasma, 99·5 ± 9·5%; and from urine, 97·1 ± 9·4%. The lower limits of sensitivity were 0·05 μg ml^{-1} for blood or plasma and 0·25 μg ml^{-1} for urine. A. R. R.

1D88. **High-pressure liquid-chromatographic analysis of carcinogenic *N*-nitroso-derivatives of piperazine resulting from drug - nitrite interactions.** Rao, G. S.; and McLennon, D. A. (Pharmacol. Lab., Div. Biochem., Am. Dental Assoc. Health Found.,

Chicago, Ill. 60611, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (1), 43–45.—The 1-nitroso- and 1,4-dinitroso-derivatives of the named anthelmintic are separated by h.p.l.c. on a column (61 cm × 2·1 mm) of silica (particle size 37 μm; Tracor Chromasep-S) operated at 24° ± 0·5°, with ethyl acetate - methanol (1 : 1) as mobile phase (1·2 ml min^{-1}) and detection at 363 nm. The method has been used in detecting the nitroso-compounds formed *in vitro* in human saliva and *in vivo* in rat stomach (after co-administration of $NaNO_2$ and piperazine); detection limits were 3 and 0·3 μg for the mono- and di-nitroso-compounds, respectively, and the corresponding calibration graphs were rectilinear up to 50 and 10 μg. B. H.

1D89. **Quantitative determination of metabolites of mercaptopurine in biological materials. I. Separation method for purine and mercaptopurine bases and nucleosides by high-pressure liquid cation-exchange chromatography.** Breter, Hans-Joachim (Physiol.-Chem. Inst., Univ. Johann Joachim Becher, Mainz, W. Germany). *Anal. Biochem.*, 1977, **80** (1), 9–19.—The separation was effected on stainless-steel columns (40 or 100 cm × 1·8 mm) packed with strongly acidic cation-exchange resin M 71 (Beckman) and operated at 50°, with ammonium formate (pH 4·6) as eluent at 13·3 and 8 ml h^{-1} for the two respective columns, this flow rate being doubled after 75 min. The eluates were monitored (with use of a variable-wavelength u.v. detector) at, *e.g.*, 254 nm for common purines, 291 nm for 6-methylthiopurines, 322 nm for 6-mercaptopurines and 342 nm for 6-mercaptoguanines. Limits of detection ranged from 15 to 30 pmol. R. A. H.

1D90. **Gas - liquid chromatographic determinations of plasma acenocoumarol [nicoumalone] levels.** Midha, K. K.; and Cooper, J. K. (Drug Res. Lab., Health Prot. Branch, Health and Welfare Canada, Tunney's Pasture, Ottawa, Ont.). *J. Pharm. Sci.*, 1977, **66** (6), 799–802.—Mix the plasma (human or canine), containing >0·5 μg of nicoumalone, with aq. internal standard [containing 15 μg of 1,2-diphenyl-4-(3-oxobutyl)pyrazolidine-3,5-dione], 3N-HCl and 1,2-dichloroethane for 30 min, centrifuge, and shake the separated organic phase with N-NaOH for 15 min. Centrifuge, acidify the separated aq. phase with 5N-HCl, and shake with dichloroethane for 20 min. Evaporate the organic phase to dryness at 85° under N, dissolve the residue in ethereal diazomethane, set aside for 10 min, and allow the ether to evaporate. Submit a soln. of the residue in CS_2 to g.l.c. at 285° on a glass column (180 cm × 2·5 mm) containing 3% of OV-11 on Chromosorb W (80 to 100 mesh), with N as carrier gas (70 ml min^{-1}) and flame ionisation detection; compare the peak heights. Recovery was quantitative, and the coeff. of variation for 0·25 μg ml^{-1} of nicoumalone (7 determinations) was 4%. A. R. R.

1D91. **Analytical toxicology: element-selective electrolytic-conductivity detection of chlorinated volatiles.** Pape, Brian E. (Dept. Pathol., Toxicol. Lab., Univ. Missouri, Columbia, Mo. 65201, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (1), 46–53.—A Tracor conductivity detector has been used in the catalytic-pyrolysis mode for detecting CH_2Cl_2, $CHCl_3$, 1,2-dichloroethane and CCl_4 separated by g.c. on a column (180 cm × 4 mm) of Porapak QS, with temp.-programming from 175° (maintained for 2 min) to 235° at 5° min^{-1} and He (25 ml min^{-1}) as carrier gas. Tests were carried out on aq. soln., urine and blood. Detector response was rectilinear for concn. from 0·1 to 100mM for the four compounds in the biological fluids. Tests were also carried out with H_2O, acetonitrile, acetone, isopropyl alcohol and ethyl acetate, and the detector response to these compounds is compared with that to the Cl-containing species. B. H.

1D92. **Rapid, direct micro-quantitative method for determination of cyclohexanone in physiological solutions and biological fluids and of the retention behaviour of volatile [organic] compounds by gas chromatography.** Hung, G. W. C. (Woodson - Tenent Lab., Hill Top Testing Services, Memphis, Tenn. 38101, U.S.A.). *Microchem. J.*, 1977, **22** (2), 157–167.—The method consists in g.l.c. (preferably at 130°) of the sample (0·1 to 5 μl) on a stainless-steel column (183 cm × 0·64 cm) packed with 30% of Carbowax 20M on Chromosorb W AW (60 to 80 mesh), with He as carrier gas (≃39 ml min^{-1}) and a flame ionisation detector. The graph of concn. *vs.* peak area for cyclohexanone (**I**) is rectilinear. Recoveries of **I** (40 to 46 p.p.m.) added to physiological saline, Chenoweth's soln. (without D-glucose), blood and urine were >96%. The retention times of 23 other volatile organic compounds are reported, the effect of column temp. on the retention of **I** is discussed briefly, and other chromatographic factors affecting accuracy are indicated. Separation of **I** from most of the volatile compounds was virtually complete when direct injection was used, so that no internal standard was needed. The sensitivity was ≃1 p.p.m. W. J. B.

1D93. **Analysis of chlordecone (Kepone) in biological specimens.** Blanke, Robert V.; Fariss, Marc W.; Griffith, Francis D., jun.; and Guzelian, Philip (Dept. Pathol., Med. Coll. Virginia, Richmond, Va. 23298, U.S.A.). *J. Anal. Toxicol.*, 1977, **1** (2), 57–62.—This pesticide ingredient (**I**) is extracted from blood, serum, plasma, urine or fat by shaking the sample, plus added [^{14}C]**I**, with H_2SO_4 and hexane - acetone (17 : 3); the extract is evaporated at 60°, and a soln. of the residue in benzene - methanol (99 : 1) is used for g.l.c. and scintillation counting to check the recovery. For g.l.c., a glass column (1·8 m × 2 mm) packed with 4% of SE-30 plus 6% of OV-210 is used, with Ar - CH_4 (19 : 1) as carrier gas (20 ml min^{-1}) and ^{63}Ni electron-capture detection; the column temp. is kept at 170° for 1 min, then programmed at 10° min^{-1} to 230°, and maintained at this value for 15 min. The detector response is rectilinear for 10 pg to 100 ng of **I** injected (*i.e.*, 5 p.p.b. to 50 p.p.m. in a 1-g sample). A modified extraction procedure is described for use with faecal samples and bile. B. H.

1D94. **Detection and determination of dichlorvos (DDVP) in toxicological analysis.** Tewari, S. N.; and Harpalani, S. P. (Toxicol. Div., Chem. Examiner's Lab., Agra, India). *Fresenius' Z. Anal. Chem.*, 1977, **285** (1), 48.—An aliquot of the homogenised sample of biological material is applied to a silica gel t.l.c. plate and the chromatogram is developed in ethyl ether - methanol (3:1). The dried layer is sprayed with ethanolic 0·05% thymol blue soln. DDVP gives a violet spot (R_F 0·36) and is determined by comparison with standard spots in the range 0·5 to 10 μg. The method is specific for DDVP, with a detection limit of 0·2 μg. Trichlorphon also gives a violet spot (R_F 0·28), but does not respond to the pyridine - alkali test. Parathion, malathion, dimethoate, diazinon, fenitrothion, demeton-*S*-methyl, fenthion, disulfoton, phosphamidon, DDT, HCH and endrin do not interfere. J. K. C.

Non-nitrogenous compounds and their derivatives

Including lipids and steroids

1D95. **Gas chromatography - mass spectrometry of metabolites in haemodialysis fluid.** Masimore, Ted L.; Veening, Hans; VandenHeuvel, W. J. A.; and Dayton, David A. (Dept. Chem., Bucknell Univ., Lewisburg, Pa. 17837, U.S.A.). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 247–257.—The filtered fluid is applied to a column of DEAE-Sephadex, from which acidic (and some neutral) compounds are eluted with pyridinium acetate. A portion of the eluate is concentrated and trimethylsilylated, and the products are submitted to temp.-programmed g.l.c. on a conventional column (3% of SE-30 on Gas-Chrom Q), with flame ionisation detection. Another portion of the eluate is similarly treated, but with a deuterated silylating reagent, and the derivatives are analysed on another column (3% of OV-1), the effluent being examined by 70-eV m.s. Chromatograms and mass spectra are discussed, and *m/e* values are listed for various compounds, including glucopyranuronolactone, citric acid, D-gluconolactone and the α- and β-epimers of D-glucose and H_3PO_4. B. H.

1D96. **Serum-glucose determination with Dextrostix and the Eyetone reflectance meter.** Hornnes, P.; and Kuhl, C. (Dept. Internal Med. T, Bispebjerg Hosp. Univ., Copenhagen, Denmark). *Acta Med. Scand.*, 1976, **200** (4), 297–299.—It is shown that the Dextrostix - Eyetone system can be used for the determination of glucose in serum by reducing the reaction time from 60 s (for analysis of whole blood) to 45 s. Orthogonal regression analysis was applied to test the correlation between concn. obtained by a glucose oxidase reference method (Trinder, *Ann. Clin. Biochem.*, 1969, **6**, 24) and the Dextrostix - Eyetone procedure with a 60-s and a 45-s reaction time. The coeff. of correlation was 0·99 for each reaction time but only for the 45-s time was the regression line close to the ideal. The modified procedure gave coeff. of variation that were almost identical to those obtained by the reference method in the ranges 10 to 100, 100 to 200 and 300 to 400 mg dl^{-1} of glucose in serum; a greater variance was obtained in the range 200 to 300 mg dl^{-1} but this did not preclude use of the method. E. K. O.

1D97. **Cerebrospinal fluid glucose measurements with Dextrostix and a reflectance meter.** Penn, Duna; Williams, Paul R.; and Adair, Robin M. (Dept. Pediatr., William Beaumont Hosp., Royal Oak, Mich., U.S.A.). *J. Pediatr.* (*St. Louis*), 1977, **90** (5), 771–773.—The usual 60-s Dextrostix test was modified by covering the active area of the reagent strip with cerebrospinal fluid and noting the colour after 45 s. For glucose concn. from 10 to 160 mg dl^{-1}, there were no significant differences between these 'Dextrostix 45' measurements (in a reflectometer) and the reference values determined by using a Beckman glucose analyser. By contrast, 'Dextrostix 60' readings deviated significantly from the reference curve. The accuracy of this Dextrostix method depends on calibration of the reflectometer at least twice daily, the use of sufficient sample to cover the tip of the reagent strip, and incubation for exactly 45 s. The glucose values are then consistent with the reference values in the concn. range stated, and for protein contents in the range 11 to 1000 mg dl^{-1}. CHEM. ABSTR.

1D98. **Paper-chromatographic estimation of fructose and myoinositol in human seminal fluid: method for evaluating seminal-vesicle and prostatic function.** Lewin, L. M.; Beer, R.; and Lunenfeld, B. (Dept. Chem. Pathol., Sackler Med. Sch., Tel Aviv Univ., Israel). *Int. J. Fertil.*, 1977, **22** (1), 48–51.—The sample (1 μl) is developed for 3 h on Whatman No. 1 paper, with 2,6-lutidine - isopropyl alcohol - H_2O (3:2:2) as mobile phase; the dried chromatogram is sprayed with alkaline $AgNO_3$ soln. (Lewin *et al.*, *Anal. Abstr.*, 1975, **28**, 3D69), and inositol (R_F 0·21) and fructose (R_F 0·42) are estimated by comparison of the intensities of the zones with those for standards (1, 2 and 4 mg ml^{-1} of fructose and 0·25, 0·5 and 1 mg ml^{-1} of inositol) developed on the same chromatogram. The use of the method in evaluating the composition of seminal fluid is discussed. B. H.

1D99. **Enzymic determination of maltose by amperometric measurement of rate of oxygen depletion.** Marshall, Linda B.; Christian, Gary D.; and Kumar, Anand (Dept. Chem., Univ. Washington, Seattle, Wash. 98195, U.S.A.). *Analyst* (*London*), 1977, **102** (1215), 424–428.—Maltose in the sample is hydrolysed by means of α-glucosidase, an impurity in glucose oxidase, and the glucose formed reacts with O in the presence of the glucose oxidase; the rate of O depletion is measured with a Clark electrode in a Beckman glucose analyser. *Procedure* —Dilute the test soln. with benzoate buffer (0·02M, pH 5·5) to give a soln. containing 0·5 to 5·0 mg ml^{-1} of maltose. Inject 25 μl of this soln. into 750 μl of glucose oxidase (140 i.u. ml^{-1} in 0·2M-PO_4^{3-} buffer of pH 6) which has been equilibrated with atmospheric O, and measure the signal max. Glucose present in the sample can be destroyed by an initial incubation, for 10 min, with purified glucose oxidase reagent; starch interference is easily corrected for by adding starch to standards, and fructose, lactose and galactose do not interfere. Good recovery of maltose from serum samples is obtained and there is no interference in the determination of blood glucose, since the max. rate for glucose is recorded before appreciable reaction from maltose has occurred. R. W.

1D100. **Spectrophotometry as a tool for assaying endotoxins.** Karamian, Narbik A.; and Waters, Paul F. (Nat. Inst. Health, Div. Res. Serv., Environ. Safety Branch, Bethesda, Md. 20014, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (5), 723–724.—Aqueous soln. of bacterial lipopolysaccharides are assayed by measurement of the absorbance at 259 nm in a 10-cm cell. Graphs of absorbance *vs.* concn. are nearly rectilinear over the ranges 0·01 to 0·05, 0·05 to 1 and 0·5 to 10 p.p.m. A. R. R.

1D101. **Sensitive radioenzymic assay for glycerol and acylglycerols.** Schneider, Peter B. (Thorndike Memorial Lab. and Nucl. Med. Unit, Beth Israel Hosp. and Harvard Med. Sch., Boston, Mass. 02215, U.S.A.). *J. Lipid Res.*, 1977, **18** (3), 396–399.—This method is based on the quantitative phosphorylation of glycerol by glycerol kinase and [γ-^{32}P]ATP. The ^{32}P-labelled glycerophosphate formed is separated by t.l.c. on cellulose, and its ^{32}P content is measured by liquid scintillation counting to give a measure of the quantity of glycerol originally present. If the glycerol has been labelled with ^{14}C or ^{3}H, the ratio of the quantity of this radionuclide to that of ^{32}P in the final glycerophosphate gives a measure of the specific activity of the

glycerol. Acylglycerols are first hydrolysed to glycerol. Rectilinear responses (c.p.m.) up to 100 nmol can be obtained. C. B.

1D102. **Determination of acetaldehyde in biological samples by head-space gas chromatography.** Eriksson, C. J. Peter; Sippel, Helmuth W.; and Forsander, Olof A. [Res. Lab., State Alcohol Monopoly (Alko), Helsinki, Finland]. *Anal. Biochem.*, 1977, **80** (1), 116–124.—Blood, liver and brain tissue samples were deproteinised with 0·6M-$HClO_4$, and portions (0·5 ml) of the supernatant soln. were transferred to 22-ml serum bottles; these were sealed and incubated for 15 min at 65°. Head-space samples were taken automatically with an electropneumatic dosing system, and the acetaldehyde was determined by g.l.c. (at 75°) on a column of 15% of polyoxyethylene glycol on Celite (60 to 100 mesh). Recoveries from the various tissues are reported. R. A. H.

1D103. **Quantitative freeze-drying of organic compounds of low relative molecular mass.** Chalmers, Ronald A. (Div. Inherited Metab. Diseases, M.R.C. Clin. Res. Centre, Harrow, Middx., England). *Methodol. Dev. Biochem.*, 1976, **5**, 121–124.—This topic is discussed with special reference to the isolation of carboxylic acids from biological samples for subsequent analysis by, *e.g.*, g.l.c. The latent heats of vaporisation are calculated by a known method, and the values so obtained are used in selecting optimum conditions for freeze-drying. Quantitative recoveries are reported for 16 commonly occurring acids. B. H.

1D104. **Anion exchangers for quantitatively extracting organic acids from urine and other fluids.** Chalmers, Ronald A. (Div. Inherited Metab. Diseases, M.R.C. Clin. Res. Centre, Harrow, Middx., England). *Methodol. Dev. Biochem.*, 1976, **5**, 135–140.—Urine or other protein-free fluid, at pH 7, is applied to a column of DEAE-Sephadex A-25, on which all compounds with pK_a values $< \simeq 5{\cdot}5$ (including anionic drugs and metabolites and conjugates with glycine and glucuronic acid) are retained. Neutral and basic compounds are washed from the column with H_2O, then the acids are eluted with pyridinium acetate soln. for freeze-drying, derivative formation and determination by g.l.c. The DEAE-Sephadex was preferred to other ion-exchange materials tested. Chromatograms are reproduced of acids from normal urine and from the urine of a patient receiving chloral hydrate. B. H.

1D105. **Chromatographic analyses of marine polyunsaturated fatty acids.** Schultz, D. M.; and Quinn, J. G. (Grad. Sch. Oceanogr., Univ. Rhode Is., Kingston, U.S.A.). *Mar. Biol.*, 1977, **40** (2), 117–120.—The effects of saponification, methylation and isolation by t.l.c. on the g.l.c. of polyunsaturated acids present in marine plant and animal species were studied. The qualitative and quantitative distribution can be determined very accurately both before and after isolation by t.l.c. C. B.

1D106. **Gas-chromatographic detection of citric and lactic acids in human prostate.** Dogru, Rezzan; Yurum, Yuda; and Erol, Demokan (Hacettepe Univ., Chem. Fac., Ankara, Turkey). *Hacettepe Bull. Nat. Sci. Eng.*, 1976, **5**, 24–31.—The tissue sample is extracted for 2 h with anhyd. ethanol, and a portion of the extract is allowed to react for 24 h at 0° with acetic anhydride and pyridine. The acetylated derivatives of lactic and citric acids are separated by g.l.c. (at 30°) of an aliquot of the reaction mixture on a column (6 ft. × 0·25 in.) packed with 1% of SE-30 on Chromosorb W (80 to 100 mesh), with N as carrier gas (30 ml min^{-1}) and flame ionisation detection. The calibration graphs (peak area) are curvilinear and cover the range up to 300 mg of each derivative. Results for normal and pathological samples are discussed. B. H.

1D107. **Oximes for high-performance liquid and electron-capture gas chromatography of prostaglandins and thromboxanes.** Fitzpatrick, F. A.; Wynalda, M. A.; and Kaiser, D. G. (Res. Lab., The Upjohn Co., Kalamazoo, Mich. 49001, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1032–1035.—The 4-nitrobenzyloxime derivatives of prostaglandin esters are useful for enhancing detection limits in h.p.l.c. (μBondapak C_{18} column, with 254-nm detection), and the pentafluorobenzyloxime derivatives are similarly useful for ^{63}Ni electron-capture g.l.c. With use of the latter derivatives, determinations are possible at the ng level, with on-column sensitivities in the pg range. G. P. C.

1D108. **Extraction of prostaglandins $F_{1\alpha}$ and $F_{2\alpha}$ from human seminal plasma.** Srivastava, K. C. (Inst. Hyg. and Social Med., Odense Univ., Denmark). *Fresenius' Z. Anal. Chem.*, 1977, **285** (1), 35–38.—Following a preliminary purification, the prostaglandins are separated by chromatography on a silica gel column, with ethyl acetate - benzene (4:1) as eluent; the eluate is concentrated by evaporation under N, and the residue is dissolved in methanol. Prostaglandins $F_{1\alpha}$ and $F_{2\alpha}$ are then separated by t.l.c. on Silica gel G with $CHCl_3$ - methanol - acetic acid - H_2O (90:9:1:0·65) as developing solvent. The zones containing 19-hydroxy-derivatives of prostaglandins A_1, A_2, B_1 and B_2 are extracted with methanol, the extracts are combined, and the prostaglandins are further separated by argentation t.l.c., with ethyl acetate - acetic acid - methanol - 2,2,4-trimethylpentane - H_2O (22:2:3:2:20) as developing solvent. Recoveries were up to 98%. J. K. C.

1D109. **Thin-layer chromatographic procedure for class separation of plant neutral lipids.** Schlotzhauer, P. F.; Ellington, J. J.; and Schepartz, A. I. (Tobacco Lab., Athens, Ga. 30604, U.S.A.). *Lipids*, 1977, **12** (2), 239–241.—Lipid samples in hexane (2 to 20 μg in 5 μl) were separated on Silica gel G by a double development, with hexane - ethyl ether (49:1) as the first solvent and hexane - ether - acetic acid (50:50:1) as the second. The lipids were located by spraying with H_2SO_4 - $K_2Cr_2O_7$ and charring at 120°, or (for preparative t.l.c.) by spraying with Rhodamine 6G (C.I. Basic Red 1) and examining under u.v. radiation. The bands were scraped off the plate, and their components were extracted (and determined) by the method of Skipski *et al.* (*Anal. Abstr.*, 1969, **16**, 2042). The lipid mixtures were separated into hydrocarbon waxes, steryl and methyl esters, mono-, di- and triglycerides, fatty acids and sterols. R. A. H.

1D110. **Quantitative analysis of plasma neutral glycosphingolipids by high-performance liquid chromatography of their perbenzoyl derivatives.** Ullman, M. David; and McCluer, Robert H. (Eunice Kennedy Shriver Center for Mental Retardation Inc., Waltham, Mass. 02154, U.S.A.). *J. Lipid Res.*, 1977, **18** (3), 371–378.—Samples containing >2·5 nmol each of mono-, di-, tri- and tetra-glycosylceramide were dried with a stream of N and then *in vacuo* over

P_2O_5, and the ceramides were benzoylated with 10% (v/v) benzoyl chloride in pyridine at 37° for 16 h. The products were separated from unconsumed reagent by extraction into hexane and, after evaporation to dryness and re-dissolution in CCl_4, were injected into a column packed with Zipax (27 μm) and operated with a rectilinear gradient of 2 to 17% of one-sixth-H_2O-saturated ethyl acetate in hexane (2 ml min^{-1} for 10 min) and detection at 280 nm. The calibration graphs were rectilinear for up to ≃2 nmol of each derivative injected, and the limit of detection was ≃70 pmol. As little as 0·5 ml of plasma could be used, and the method was applied to the analysis of erythrocyte and plasma glycolipids. C. B.

1D111. **Determination of less than a nanomole of cerebrosides by high-performance liquid chromatography with gradient-elution analysis.** Jungalwala, Firoze B.; Hayes, Lyle; and McCluer, Robert H. (Eunice Kennedy Shriver Center for Mental Retardation Inc., Waltham, Mass. 02154, U.S.A.). *J. Lipid Res.*, 1977, **18** (3), 285–292.—This method, which is more sensitive than that involving isocratic elution (McCluer and Evans, *Anal. Abstr.*, 1977, **32**, 2D39), was used for the separation of 0·5 to 10-nmol amounts of benzoylated hydroxy- and non-hydroxy-fatty-acid cerebrosides. After being dried *in vacuo* over P_2O_5, the samples were treated with 10% (v/v) benzoyl chloride in pyridine at 60° for 1 h. The products were separated from unconsumed reagent by extraction into hexane and, after evaporation to dryness and re-dissolution in CCl_4, portions were injected into a column packed with Zipax (27 μm) and operated with a rectilinear gradient of 2·8 to 5·5% of 1,4-dioxan in hexane (4 ml min^{-1} for 3 min) and detection at 230 nm. No prior purification was necessary. The detector response was the same for all derivatives, the calibration graph was rectilinear for up to ≃2 nmol injected, and the limit of detection was ≃10 pmol. C. B.

1D112. **Thin-layer chromatography and liquid chromatography. I. Application of thin-layer chromatography to separation of steroids by adsorption liquid chromatography.** Okumura, Tamotsu (Res. Lab., Shionogi & Co. Ltd., Fukushima-ku, Osaka-shi, Osaka, Japan). *Bunseki Kagaku*, 1977, **26** (6), 396–401 (in Japanese).—An investigation of the column chromatography on silica gel of several 'difficult-to-separate' stereoisomers is reported. The suitability of solvent systems was first studied by t.l.c. on silica gel, but seven systems that gave good separation in t.l.c. were inadequate when used for column chromatography, for the same solvent ratio. For sharp separation the solvent strength in the binary mixtures used for t.l.c. had to be decreased. For example, the ratio in the $CHCl_3$ - hexane system used for the separation of 5,6-epoxycholesteryl benzoates had to be reduced from 4 : 1 to 1 : 1.

1D113. **Simple method for preparation of cholesteryl esters [and their subsequent separation].** Prabhudesai, A. V. (Dept. Biochem., Maharashtra Assoc. Cult. Sci., Poona, India). *Lipids*, 1977, **12** (2), 242–244.—A mixture (450 mg) of, *e.g.*, oleic, linoleic, dodecanoic and stearic acids was treated with cholesterol (500 mg) in anhyd. benzene at 0°, $SOCl_2$ (0·25 ml) was added to the soln. dropwise, and, after flushing with CO_2, this mixture was either kept overnight at 25° to 27° or heated under reflux at 72° ± 2° for 90 min. The reaction product was washed with H_2O (2 × 50 ml), and the benzene phase was dried over Na_2SO_4 and subjected to t.l.c. on Silica gel G with light petroleum - ethyl ether - acetic acid (90 : 10 : 1) as developing solvent. A mixture of unsaturated cholesteryl esters was separated by the same procedure, and components were further purified by t.l.c. on Silica gel G impregnated with 5% of $AgNO_3$, with light petroleum (boiling-range 40° to 60°) - ether (9 : 1) as solvent. Good yields (>90%) of the esters were obtained. R. A. H.

1D114. **Gas-chromatographic separation of cholesteryl esters of fatty acids of different degrees of unsaturation.** Takagi, Toru; Sakai, Akira; Itabashi, Yutaka; and Hayashi, Kenji (Dept. Chem., Fac. Fish., Hokkaido Univ., Hakodate, Japan). *Lipids*, 1977, **12** (2), 228–232.—The cholesteryl esters of the fatty acids derived from linseed oil, pig-liver lipids and Japanese anchovy oil were prepared by heating the methyl esters of the acids (1 g) with cholesteryl acetate (1·5 g), dioxan (2 ml) and K t-butoxide (50 mg) at 70° for 1 h under N. The products were separated by g.l.c. on dual glass columns (0·5 m × 3 mm) packed with 5% of Silar 10C on Gas-Chrom Q (100 to 120 mesh) and operated at 270° or temp.-programmed from 240° to 270° (at 2° min^{-1}); N was used as carrier gas (40 ml min^{-1}) and detection was by flame ionisation. The peaks were identified by comparison with those of standards of synthetic cholesteryl esters, and their areas were measured by use of a digital integrator. Changes in retention time with chain length and degree of unsaturation are recorded for various esters. R. A. H.

1D115. **Gas-chromatographic separation of plant sterols.** Homberg, E. (Bundesanst. Fettforsch., Muenster, W. Germany). *Fette, Seifen, Anstrichm.*, 1977, **79** (6), 234–241 (in German).—Optimum conditions for the g.l.c. separation and identification of such sterols are reviewed, with particular reference to the choice of support and stationary phase (suitable materials are listed, with their McReynolds constants and max. operating temp.), the preparation and characteristics of the column, the choice of derivatives, the oven temp., and the detector. (66 references.) R. F. S.

1D116. **Mass spectra of sterically crowded trialkylsilyl ether derivatives of steroids.** Quilliam, M. A.; and Westmore, J. B. (Dept. Chem., Univ. Manitoba, Winnipeg, Canada). *Steroids*, 1977, **29** (5), 579–611.—A study is presented of the mass spectra of t-butyldimethylsilyl, t-butyl(cyclobutyl)silyl and cyclobutylisopropylsilyl derivatives of cholesterol, epicholesterol, androsterone, epiandrosterone, androstanolone, dehydroepiandrosterone, testosterone, 5β-pregnane-3α,20α-diol and 5α-androst-2-ene-3β,17β-diol. The fragmentation mechanism of these derivatives is discussed, and it is concluded that they are useful compounds for selected-ion monitoring, particularly for distinguishing between epimers. B. H.

1D117. **Sterically crowded trialkylsilyl ether derivatives for analysis of steroid metabolites.** Quilliam, M. A.; Templeton, J. F.; and Westmore, J. B. (Dept. Chem., Univ. Manitoba, Winnipeg, Canada). *Steroids*, 1977, **29** (5), 613–626.—The advantages of such derivatives over more conventional trimethylsilyl ether derivatives for use in studies by t.l.c., g.l.c. and g.l.c. - m.s. are pointed out, and the use of

t-butyldimethylsilyl, t-butylcyclobutylsilyl and cyclobutylisopropylsilyl derivatives in studying metabolites of 2α,3α-cyclopropano-5α-androstan-17β-ol in rabbit urine is discussed B. H.

1D118. **The Zimmermann reaction and a correction for irrelevant absorption.** Feldkamp, C. S.; Watkins, R. E.; Thibert, R. J.; Epstein, E.; and Zak, B. (Dept. Pathol., Wayne State Univ. School Med., Detroit, Mich. 48201, U.S.A.). *Microchem. J.*, 1977, **22** (2), 201–209.—Two similar modifications of this reaction were studied to determine whether or not the Allen correction could be accurate when pure steroids were compared with dehydroisoandrosterone as calibration standard. In the procedure of Egloff *et al.* (*Clin. Chim. Acta*, 1975, **59**, 147), involving widely divergent ϵ values, peak max. wavelengths and spectral shapes, the Allen correction led to inaccuracy when applied to pure compounds. In the other procedure (Watkins *et al.*, *Microchem. J.*, 1976, **21**, 466), in which the ϵ values and fine structures were similar, but band-widths were dissimilar, peak measurements yielded higher accuracy than was attained by applying the Allen correction. This correction was valid only for procedures in which calibration standard and sample gave spectra showing greater uniformity than in the two procedures studied. W. J. B.

1D119. **Improved method for analysis of oestrogenic steroids in pregnancy urine by high-performance liquid chromatography.** Dolphin, R. J.; and Pergande, P. J. (Mullard Res. Lab., Redhill, Surrey, England). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 267–274.—The sample is treated with conc. HCl and the liberated steroids are extracted into ethyl ether (*cf.* Dolphin, *J. Chromatogr.*, 1973, **83**, 421); the extract is concentrated and subjected to h.p.l.c. on a column (15 cm × 4·9 mm) of Partisil-5 (7 μm), with heptane - ethanol (19 : 1) as mobile phase (5 ml min^{-1}) and detection at 280 nm. A second extract is evaporated to dryness, and a soln. of the residue in methanol - aq. 0·1% ammonium carbonate (11 : 9) (solvent *A*) is subjected to h.p.l.c. on a column (25 cm × 4·6 mm) of Partisil-10 ODS, with solvent *A* as mobile phase. Chromatograms from both systems are reproduced, and the advantages and limitations of the two systems, which complement each other, are discussed. B. H.

1D120. **Isocratic separation of oestrogen conjugates on diethylaminoethyl-Sephadex.** Musey, P. I.; Collins, D. C.; and Preedy, J. R. K. (Div. Endocrinol., Emory Univ. Sch. Med., Atlanta, Ga. 30303, U.S.A.). *Steroids*, 1977, **29** (5), 657–668.—The glucuronides of oestrone and 17β-oestradiol have been separated by elution with 0·05M-NaCl from a column (60 cm × 5 mm) of DEAE-Sephadex A-25 or LH-20; for the 3-sulphate conjugates, 0·2M- or 0·25M-NaCl is preferred as eluent. The advantages of this technique over gradient elution are discussed. B. H.

1D121. **Use of an ion-exchange resin for rapid extraction in the determination of urinary oestrogens and pregnanediol in pregnancy by colorimetry or gas chromatography.** Pierrat, G.; Prost, M.; Lavoue, G.; and Lumbroso, C. (Orimbio S. A., Paris). *Ann. Biol. Clin.* (*Paris*), 1976, **34** (6), 423–430 (in French).—The sample, diluted (if necessary) to 10 ml, is hydrolysed at pH 5·2 (acetate buffer) with a β-glucuronidase - arylsulphatase preparation, mixed with the internal standards (5β-pregnane-3α,17α,20β-triol and 16,17-epioestriol), then adjusted to pH 10 to 11 and applied to a column (3 cm × 1 cm) of Bio-Rad AG 1-X2 resin (acetate form; 50 to 150 mesh) previously treated with 3 ml of M-NaOH and washed with water. The percolate is rejected, the column is washed with water (5 ml), and the pregnane-diol and -triols are eluted with methanol (5 ml); the oestrogens are eluted separately with methanol (5 ml) after washing the column with water (5 ml) and 2M-HCl (5 ml). The two fractions are evaporated separately at $<60°$ under N or *in vacuo*. Pregnanediol is determined by g.l.c., as the trimethylsilyl ether derivative, on a 3-m column of 1% of OV-1 on Chromosorb W, with temp.-programming from 210° to 260° at 2° min^{-1}. The oestrogens are determined either colorimetrically (Ittrich, *Anal. Abstr.*, 1959, **6**, 3136) or by g.l.c. on the OV-1 column temp.-programmed from 200°. E. J. H. B.

1D122. **Conjugation of testosterone with horseradish peroxidase, and a sensitive enzyme assay for the conjugate.** Rajkowski, K. M.; Cittanova, N.; Desfosses, B.; and Jayle, M. F. (Lab. Chim. Biol., Fac. Med., rue des Saints Peres, Paris, France). *Steroids*, 1977, **29** (5), 701–713.—In connection with the determination of testosterone in plasma by enzyme immunoassay, a procedure is described for preparing the testosterone 3-carboxymethyloxime - peroxidase conjugate by the conventional 'mixed anhydride' method. To determine 1 to 5 ng ml^{-1} of the conjugate, the sample is incubated for 2 h at pH 6 (0·02M-phosphate buffer) with 0·45mM-H_2O_2 and 2·5mM-5-aminosalicylic acid (as H-donor) in the presence of 0·1M-$(NH_4)_2SO_4$; the product is measured at 490 nm. Polystyrene apparatus should not be used. Solutions (930 ng ml^{-1}) of the conjugate are stable for at least a month, and the freeze-dried conjugate can be stored at −15° for at least a week with loss of $<2\%$ of its activity. B. H.

1D123. **Steroids. V. Rapid thin-layer chromatographic assay for dehydroepiandrosterone in urine.** Hakl, Jiri (Dept. Med. Chem., J. E. Purkyne Univ., Brno, Czechoslovakia). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 317–320.—Sulphate conjugates are hydrolysed by heating the sample at 100° with acetate buffer (pH ≃4·5); the mixture is well cooled, then the dehydroepiandrosterone (**I**) is extracted into cyclohexane - ethyl ether. The organic layer is washed with NaOH soln. and with H_2O and then evaporated to dryness. The residue is heated at 70° to 80° with a 2,4-dinitrophenylhydrazine reagent and trichloroacetic acid, the mixture is slowly evaporated to dryness, and a soln. of the residue in benzene is submitted to t.l.c. on Silufol silica gel, with $CHCl_3$ - acetone (19 : 1) as mobile phase (two developments; R_F 0·26 and 0·46, respectively, for the derivative of **I**). The appropriate zone is extracted with methanol, and **I** is determined from the absorbance of the extract at 366 nm. The sensitivity is ≃0·1 mg l^{-1} of **I** in the sample. (For Part IV see *Anal. Abstr.*, 1977, **33**, 4D74.) B. H.

1D124. **Specific enzyme immunoassay for progesterone in human plasma.** Joyce, B. G.; Read, G.; and Fahmy, D. R. (Tenovus Inst. Cancer Res., Welsh Natl. Sch. Med., Cardiff, Wales). *J. Endocrinol.*, 1976, **71** (7), 53P–54P.—An antiserum raised against the progesterone - bovine serum albumin conjugate is coupled to Sepharose 4B by the accepted CNBr procedure, and the product is incubated with progesterone-'labelled' peroxidase and progesterone in an extract of the sample. Free and bound forms of the steroid are separated by centrifugation, and the resulting pellet is analysed

for the enzyme by reaction with *o*-phenylenediamine and H_2O_2; the amount of reaction product (measured at 492 nm) is inversely proportional to the progesterone content of the sample. The sensitivity is 93 pg, and 100 assays can be carried out in 1 day. Only three steroids cross-react; of these, only 5α-pregnanedione interferes significantly. B. H.

1D125. **Determination of aldosterone in biological fluids.** James, V. H. T.; and Wilson, G. A. (Dept. Chem. Pathol., St. Mary's Hosp. Med. Sch., London, W.2, England). *Methodol. Dev. Biochem.*, 1976, **5**, 149–158.—Plasma plus [^{3}H]aldosterone is extracted with CH_2Cl_2, the filtered extract is applied to a column of silica gel, the column is washed with CH_2Cl_2 and with CH_2Cl_2 - methanol (50 : 1), then aldosterone is eluted with CH_2Cl_2 - methanol (100 : 9). A portion of the eluate is submitted to radio-immunoassay (*cf.* MacKenzie and Clements, *J. Clin. Endocrinol.*, 1974, **38**, 622), with use of an antiserum raised against an aldosterone 3-oxime conjugate and separation of free and bound radioactivity with dextran-coated charcoal. A modification of the procedure is applied to urine. The sensitivity is ≃5 pg. B. H.

1D126. **Determination of corticoids and related steroid analogues by high-performance liquid chromatography.** Tymes, N. W. (Food and Drug Adm., Baltimore, Md. 21201, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (5), 151–155.—Retention values, relative to acetone, are reported for 28 compounds (corticoids and related steroid analogues plus methyl and propyl hydroxybenzoate) on Bondapak Phenyl/Corasil, Bondapak C_{18}/Corasil and μBondapak C_{18}, with methanol - water or acetonitrile - water as mobile phase. Each eluate is monitored at 254 nm, and the detector response is typically rectilinear from 0·25 to 3·0 μg. Plate heights as low as 0·22 mm are achieved on the microparticulate column.

M. J. Sl.

1D127. **High-performance liquid chromatographic determination of free hydrocortisone in urine.** Schwedt, G.; Bussemas, H. H.; and Lippmann, C. (Inst. Arbeitsphysiol., Univ. Dortmund, W. Germany). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 259–266 (in German).—Urine (at pH 6) is extracted with $CHCl_3$, the extract is washed with 0·1M-KOH and with H_2O and then evaporated to dryness, and a soln. of the residue in $CHCl_3$ is subjected to h.p.l.c. at 22° on a column (25 cm × 2·1 mm) of Zorbax-Sil polar (5 to 7 μm), with $CHCl_3$ - methanol - H_2O (983 : 15 : 2) as mobile phase (0·6 ml min^{-1}) and detection at 254 nm. The chromatographic stage takes 10 to 15 min, and the limit of determination is 3 ng. B. H.

1D128. **Spectrophotometric method for determination of total steroidal sapogenin.** Baccou, J. C.; Lambert, F.; and Sauvaire, Y. (Lab. Physiol. Veg., Univ. Sci. et Tech. Languedoc, Montpellier, France). *Analyst* (*London*), 1977, **102** (1215), 458–465.—*Determination of sapogenin*—Treat 2 ml of an ethyl acetate soln. of up to 40 μg of steroidal sapogenin with 1 ml of a 0·5% soln. of anisaldehyde in ethyl acetate and 1 ml of conc. H_2SO_4 - ethyl acetate (1 : 1). Heat the stirred soln. at 60° in a water bath for 20 min, cool in a water bath at room-temp. for 10 min and measure the absorbance at 430 nm. Alternatively, use conc. H_2SO_4 instead of the H_2SO_4 - ethyl acetate and omit the heating step. *Determination of saponin*—Remove the solvent, at 100°, from an alcoholic soln. containing saponin equiv. to <40 μg of sapogenin. After cooling, add 2 ml of ethyl acetate and proceed as above. There is virtually no interference from sugars, sterols, fatty acids or vegetable oils, and the method proved satisfactory for 13 sapogenins examined, whether in the free or bound state. Identical, rectilinear calibration graphs were obtained for diosgenin, tigogenin, smilagenin and hecogenin. R. W.

1D129. **Analysis of metabolic profiles of bile acids in urine using a lipophilic anion exchanger and computerised gas - liquid chromatography/mass spectrometry.** Alme, B.; Bremmelgaard, A.; Sjovall, J.; and Thomassen, P. (Dept. Chem., Karolinska Inst., Stockholm, Sweden). *J. Lipid Res.*, 1977, **18** (3), 339–362.—Urine was acidified with 4M-HCl, and bile acids were retained on an Amberlite XAD-2 column. The resulting bile salts were converted into the acids on an Amberlyst A-15 column and were separated into groups with use of a column of diethylaminohydroxypropyl-Sephadex LH-20 and ammonium acetate soln. of increasing concn. as eluents. After solvolysis of the mono-, di- and trisulphates with dil. H_2SO_4 soln. in ethyl acetate - ethanol (9 : 1) at 39° for 16 h and hydrolysis of the products with 15% NaOH soln. in aq. 50% ethanol at ≃115° for 10 h, and hydrolysis of the glycine- and taurine-conjugated bile acids in the same way, the products were purified on another column of diethylaminohydroxypropyl-Sephadex LH-20. The resulting bile acids, and the original unconjugated and neutral ones, were converted into the trimethylsilyl ether derivatives of their methyl esters for g.l.c. - m.s. with repetitive magnetic scanning over the *m/e* range 0 to 750. About 30 bile acids could be identified fully or partially. The results indicated the existence of selective mechanisms for sulphation, hydroxylation, and renal elimination of the conjugates. C. B.

Nitrogenous compounds

Including amino-acids, peptides, proteins, pigments, and nucleic acids and their constituents

1D130. **Chromatography of biogenic amines. I. Generally applicable separation and detection methods.** Seiler, Nikolaus (Max Planck Inst. Brain Res., Neurochem. Unit, Frankfurt a. M., W. Germany). *J. Chromatogr.*, 1977, **143** (3); *Biomed. Appl.*, **1** (3), 221–246.—The topics discussed in this review are—(*i*) chromatography and electrophoresis of free amines on paper and thin layers of adsorbents; (*ii*) ion-exchange chromatography; and (*iii*) the formation of coloured derivatives for t.l.c. and of fluorescent derivatives for t.l.c. and column chromatography. (265 references.) B. H.

1D131. **New method for qualitative and quantitative determination of di- and poly-amines in animal tissues by gas - liquid chromatography.** Beninati, Simone; Sartori, Claudia; and Argento-Ceru, Maria Paola (Inst. Histol. and Embryol., Fac. Sci., Univ., Rome, Italy). *Anal. Biochem.*, 1977, **80** (1), 101–107.—Tissue homogenates (10 ml) were treated with 6% $HClO_4$ soln. (10 ml) and centrifuged. The residual material was then washed with 3% $HClO_4$ (2 × 5 ml), the supernatant liquid was made alkaline with 4M-KOH, and, after separation of the pptd. $KClO_4$, the soln. was satd. with Na_2SO_4 and extracted with butanol. The dried extract containing the amines was subjected to g.l.c. on a borosilicate-glass column (180 cm × 3 mm) containing Corning glass beads

(60 to 80 mesh) coated with 1% of KOH and 0·4% of Carbowax 20M and temp.-programmed from 100° to 200° (at 10° min^{-1}); N was used as carrier gas (12 ml min^{-1}), detection was by H-flame ionisation, and the internal standards were dexamphetamine and benzoamphetamine. Good separation of cadaverine, putrescine, spermidine and spermine was achieved, and their recoveries were >80%.

R. A. H.

1D132. **Determination of acetylcholine, choline and butyrylcholine by pyrolysis - gas chromatography - mass spectrometry.** Iwai, Yumiko; Yamamoto, Eisuke; and Muteki, Takesuke (Sch. Med., Kurume Univ., Japan). *Kurume Igakkai Zasshi*, 1976, **39** (12), 1216–1223 (in Japanese).—Acetylcholine, choline and butyrylcholine dissolved in methanol were determined simultaneously without pre-treatment. The compounds, after pyrolytic *N*-demethylation at 400° to 450° for 10 s, were separated on a column (2 m × 2 mm) packed with 20% of poly(diethylene glycol succinate) on Gas-Chrom Q, with temp.-programming from 70° to 300° at 4° min^{-1}, and identified by m.s. Detection limits were in the ng range. Succinylcholine chloride and succinylmonocholine chloride could not be separated, as they formed the same pyrolysis product.

CHEM. ABSTR.

1D133. **Analysis of acidic monoamine metabolites by gas chromatography - mass spectrometry.** Godse, Damodar; Warsh, Jerry J.; and Stancer, Harvey C. (Sect. Neurochem., Clark Inst. Psychiatry, Univ. Toronto, Ont., Canada). *Anal. Chem.*, 1977, **49** (7), 915–918.—Derivatisation of such metabolites (*viz*, 5-hydroxyindol-3-ylacetic acid and 4-hydroxy-3-methoxymandelic acid) with fluorinated and non-fluorinated alcohols and pentafluoropropionic anhydride yielded several products that were analysed by g.l.c. - m.s. The column (1·5 m × 2 mm) was packed with 3% of OV-17 on Gas-Chrom Q (100 to 120 mesh) and (depending on the reaction studied) operated at 115° or 130° or temp.-programmed (10° min^{-1}) from 145° to 200° after isothermal operation at 145° for 1 min; He was used as carrier gas (20 ml min^{-1}) and detection was by means of a 70-eV m.s. system equipped with a programmable multiple-ion monitor. Optimum conditions for the derivatisation reaction are discussed.

G. P. C.

1D134. **Selective introduction of trimethylsilyl groups in tryptamines: preparation of *N*1-trimethylsilyl derivatives of tryptamines.** Narasimhachari, N.; and Leiner, K. (Galesburg State Res. Hosp., Ill. 61401, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (5), 181–184.—Indolylethylamines were treated with trifluorobis(trimethylsilyl)acetamide plus 1% of chlorotrimethylsilane at 100° in a sealed tube for 15 min. Mass spectra of the products showed that the trimethylsilyl groups were substituted on the indolic N atoms. Confirmation of this was achieved by converting these compounds into their isothiocyanato-derivatives (by treating with CS_2 in ethyl acetate for 30 min). Retention times are given for tryptamine and 5-hydroxy-, 5-methoxy- and α-methyl-tryptamine and their trimethylsilyl derivatives on SE-30, OV-17 and OV-225. The mass spectra of the derivatives are reproduced.

M. J. SL.

1D135. **Serotonin [5-hydroxytryptamine] in blood platelets: studies on a simple assay.** Goldman, J.; and Thibert, R. J. (Dept. Chem., Univ. Windsor, Ont., Canada). *Microchem. J.*, 1977, **22** (1), 85–91.—Platelet pellets, prepared from platelet-rich plasma, were vigorously shaken with H_2O and the resulting lysates were mixed with edta-treated plasma, $ZnSO_4$ and NaOH. After centrifuging, the 5-hydroxytryptamine in the supernatant soln. (pH ≃6·5) was determined spectrofluorimetrically at 335 nm with excitation at 301 nm. The fluorescence was directly related to concn. in the range 0·001 to 1·0 μg ml^{-1}, with no appreciable variation between pH 2 and 10. Coefficients of variation were <10%. There was no interference from a 10-fold excess of tryptophan or *NN*-dimethyltryptamine, but equivalent amounts of 5-hydroxytryptophan and 5-hydroxyindol-3-ylacetic acid exhibit 10% and 60% positive interference, respectively.

T. G. A.

1D136. **Newer method for determination of plasma histamine—an improved fluorimetric assay.** Suzuki, O.; Ishii, K.; and Tsuchiya, M. (Sch. Med., Keio Univ., Tokyo, Japan). *Biochem. Exp. Biol.*, 1976, **12** (2), 185–191.—The method is essentially the same as that of Shore *et al.* (*cf. Anal. Abstr.*, 1960, **7**, 3424), but involves recrystallisation of phthalaldehyde to improve the fluorescence intensity of the reaction (which is effected at 0° for 45 to 60 min), use of citric acid to stabilise the fluorescence intensity, and use of a Cellex-P column to separate histidine and spermidine completely from histamine. The average histamine level in 20 normal subjects was 7·05 ± 1·09 μg l^{-1}.

CHEM. ABSTR.

1D137. **Modified electron-capture gas - liquid chromatographic assay for salsolinol [1-methyl-1,2,3,4-tetrahydroisoquinoline-6,7-diol] in brain tissue.** O'Neill, Patrick J.; and Rahwan, Ralf G. (Div. Pharmacol., Coll. Pharm., State Univ., Columbus, Ohio 43210, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 893–895.—The method was devised for use in studying the *in vivo* formation of tetrahydroisoquinoline alkaloids from dopamine. *Procedure*—Homogenise a single mouse brain (containing 4 to 400 ng of salsolinol) with satd. NaCl soln. in 0·05N-oxalic acid and 25% butanol soln. in isopropyl alcohol, add tyramine as internal standard, centrifuge at 3000 *g* for 5 min, and shake 3 ml of the upper phase with hexane and 0·5M-Na_2HPO_4 of pH 6·5. Centrifuge, saturate 0·5 ml of the lower phase with NaCl, and extract it with the butanol soln. Evaporate the extract to dryness under N, add acetonitrile and heptafluorobutyric anhydride to the residue, and, after 30 min at room temp., evaporate to dryness, and dissolve the residue in benzene. Wash the soln. with satd. aq. $Na_2B_4O_7$, reject the washings, then immediately submit a portion to g.l.c. at 150° on a glass column (6 ft. × 2 mm) containing 3% of OV-17 on Gas-Chrom Q (100 to 120 mesh), with Ar - CH_4 (19 : 1) as carrier gas (20 ml min^{-1}) and electron-capture detection (^{63}Ni). Compare the peak heights.

A. R. R.

1D138. **5-Isothiocyanato-1,8-naphthalenedicarboxy-4-methylphenylimide {5-isothiocyanato-1,3-dioxo-2-*p*-tolyl-2,3-dihydro-1*H*-benz[*de*]isoquinoline}, a fluorescence reagent for compounds containing amino-groups.** Khalaf, Hosni; and Rimpler, Manfred (Inst. Klin. Biochem. Physiol. Chem., Med. Hochsch. Hannover, W. Germany). *Hoppe-Seyler's Z. Physiol. Chem.*, 1977, **358** (4), 505–511 (in German).—This compound (synthesis described) is recommended as a reagent for the qualitative and quantitative determination of such compounds as amino-acids, proteins and amines.

CHEM. ABSTR.

1D139. **Thin-layer chromatographic resolution of the racemates of some amino-acids.** Bach, Karl; and Haas, Hermann Josef (Fachricht. Physiol Chem., Fachber. 3, Univ. Saarland, Homburg, W. Germany). *J. Chromatogr.*, 1977, **136** (1), 186–188 (in German).—Racemic mixtures of amino-acids and sulphonated amino-acids were resolved by t.l.c. on Cellulose F_{254} aluminium foils (Merck) with various developing solvents, and were detected with ninhydrin. In each case the D-isomer had a higher R_F value than the L-isomer. A very good separation of DL-*p*-tyrosine-3-sulphonic acid was obtained, but DL-*β*-phenylserine and DL-histidine (which are separable by paper chromatography) were not resolved. R. F. S.

1D140. **Rapid gas-chromatographic separation of amino-acid enantiomers with a novel chiral stationary phase.** Frank, Hartmut; Nicholson, Graeme J.; and Bayer, Ernst (Inst. Org. Chem., Auf der Morgenstelle, Tuebingen, W. Germany). *J. Chromatogr. Sci.*, 1977, **15** (5), 174–176.—*N*-Pentafluoropropionyl-DL-amino-acid isopropyl esters were prepared by heating the enantiomeric amino-acids for 1 h at 100° with 2M-HCl in isopropyl alcohol, then evaporating the reagent and dissolving the residues in CH_2Cl_2 - pentafluoropropionic anhydride. After 1 h at room temp., the solvents were evaporated and the residues were dissolved in $CHCl_3$. Portions of these soln. were subjected to g.l.c. on a glass column (18 m × 0·33 mm) which had been treated with colloidal silicic acid or Aerosil and coated with 0·15% of stationary phase in $CHCl_3$. The phase was prepared by coupling L-valine t-butylamide to a copolymer of dimethylsiloxane and 'carboxyalkylmethylsiloxane'. After 3 min at 90°, the temp. was raised at 4° min^{-1} to 170° and maintained at 170° for 10 min. With this temp. programme and a mixture of 17 amino-acids, 14 enantiomers were resolved but D- and L-proline, D-isoleucine and L-alloisoleucine, and L-threonine and D-alloleucine were not. M. J. Sl.

1D141. **Amino-acid analysis by gas - liquid chromatography of *N*-heptafluorobutyryl isobutyl esters. Complete resolution using a support-coated open-tubular capillary column.** Pearce, R. J. (CSIRO, Dairy Res. Lab., Highett, Vic., Australia). *J. Chromatogr.*, 1977, **136** (1), 113–126.—Samples containing amino-acids (25 to 250 nmol of each) were freeze-dried and residual H_2O was removed azeotropically by evaporation with dry CH_2Cl_2 (100 μl). Isobutyl alcohol - 3N-HCl (≃ 100 μl) was added and the mixture was heated at 110° for 30 min under N. Unconsumed reagent was evaporated and the residue was dried azeotropically as above. Dry ethyl acetate (25 μl) containing BHT (equimolar with respect to each amino-acid) and heptafluorobutyric anhydride (10 μl) were added, and the mixture was heated at 110° for 10 min under N. To obtain a volatile histidine derivative the product was evaporated as above, diethyl oxydiformate (5 μl) and ethyl acetate (30 μl) were added, and the mixture was heated at 110° for 5 min under N. In either case the soln. was evaporated nearly to dryness and the residue was dissolved in ethyl acetate (30 μl). The soln. was then subjected to g.l.c. on a glass column (60 m × 0·5 mm) coated with SE-30, temp.-programmed from 100° to 250° at 6° min^{-1} and maintained at 250° for 5 min, with He as carrier gas (4 ml min^{-1}) and a flame ionisation detector. M. J. Sl.

1D142. **Ultra-micro gas-chromatographic determination of amino-acids using glass open-tubular columns and a nitrogen-selective detector.** Adams, R. F.; Vandemark, F. L.; and Schmidt, G. J. (Perkin-Elmer Corp., Norwalk, Conn. 06856, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (2), 63–68.—Amino-acids in biological samples are isolated by adsorption on Dowex 50-X8 (H^+ form), the resin is washed to remove anions and non-ionic material, then the amino-acids are eluted with 2N-NH_3 and the eluate is evaporated to dryness. 2,2-Dimethoxypropane (10 μl) followed by propanolic 8M-HCl (200 μl) is added and the mixture is heated at 110° for 15 min in a closed tube to form propyl derivatives of the amino-acids. The mixture is cooled, evaporated under N at 75° and treated with acylation reagent [acetone - triethylamine - acetic anhydride (5 : 2 : 1; 200 μl)] and heated at 60° for 1 min. The soln. is evaporated to dryness and the residue is dissolved in ethyl acetate (10 μl). An aliquot of the soln. is analysed by g.l.c. on a glass capillary column (50 m × 0·27 mm) coated with Carbowax 20M - Silar 5 CP (1 : 1) and temp.-programmed from 110° to 190° at 8 min^{-1}, then maintained at 250° for 10 min, with He as carrier gas (0·75 ml min^{-1}) and a thermionic detector. The detector is 80 to 180 times more sensitive than a flame ionisation detector, and the results are reproducible. M. J. Sa.

1D143. **Micro-determination of α-amino-acids by colorimetry and atomic-absorption spectrophotometry.** Gawargious, Y. A.; Besada, Amir; and Hassouna, M. E. M. (Microanal. Res. Lab., Natl. Res. Centre, Dokki, Cairo, Egypt). *Microchem. J.*, 1977, **22** (1), 96–102.—To a soln. containing an amino-acid (0·5 to 3·0 mg) was added a suspension of copper phosphate (Schroeder *et al.*, *Anal. Chem.*, 1950, **22**, 760) and the mixture was centrifuged. Copper in the supernatant soln. was determined either by a.a.s. at 324·8 nm or colorimetrically by the addition of KCN, molybdophosphoric acid and HCl and measurement at 725 nm of the absorbance due to molybdenum blue. The α-amino-group content was calculated by assuming a Cu-to-amino-acid ratio of 1 : 2. Results obtained for nine amino-acids by both procedures are listed. T. G. A.

1D144. **Fractionation of amino-acids by low-voltage electrophoresis on cellulose acetate gel. (Its application to separation of urinary amino-acids.)** Castagnino, Juan Miguel; and Foti, Angel (Fac. Cienc. Exactas Naturale, Univ. Buenos Aires, Argentina). *Acta Bioquim. Clin. Latinoam.*, 1976, **10** (1), 49–52 (in Spanish).—The urinary amino-acids were separated by electrophoresis on such gels in acetate buffer medium of pH 2·2, then were located on the electropherogram with, *e.g.*, ninhydrin reagent and determined by densitometry. Chem. Abstr.

1D145. **Critical role of acetic anhydride in rapid determination of tryptophan.** Makinde, Michael A.; Hare, Lynne B.; and Lachance, Paul A. (Dept. Food Sci., Cook Coll., Rutgers–State Univ. New Jersey, New Brunswick, N.J. 08903, U.S.A.). *Nutr. Rep. Int.*, 1976, **14** (5), 547–552.—A method is described for determining the optimum addition of acetic anhydride (**I**) to the acetic acid - $FeCl_3$ - H_2SO_4 reagent of Dalby and Tsai (*Anal. Abstr.*, 1975, **29**, 3D92). Reagent containing various proportions of **I** was added to tryptophan soln. (0 to 120 μg ml^{-1}), and the colour formed was measured at 545 nm. Results were statistically analysed and a 'response surface plot' was drawn; from this, the optimum level of **I** could be chosen. E. A.

Vitamins; enzymes

1D146. **Determination of folic acid by radio-assay.** Heilmann, E.; and Boenninghoff, E. (Med. Poliklinik, Univ. Muenster, W. Germany). *Diagnostik*, 1976, **9** (10), 347–349 (in German).—The assay of folic acid (**I**) in plasma is based on inhibition by **I** of the binding of ^{3}H-labelled **I** to a **I**-specific protein obtained from milk. A commercial test kit (Firma Schwarz/Mann, Heidelberg) is used. Reproducible results were obtained in 600 assays on plasma from healthy men and women receiving tablets each containing 0·25 mg of **I**. Results for normal subjects not receiving **I** ranged from 3·2 to 20·2 ng ml^{-1} (average 6·8 ng ml^{-1}). The procedure is suitable for differentiating between anaemias due to deficiency of cyanocobalamin and of **I**. F. R. B.

1D147. **Comparison of toadfish-serum competitive binding and microbiological assays of vitamin B_{12} [cyanocobalamin].** Buchanan, Julia W.; McIntyre, Patricia A.; Scheffel, Ursula; and Wagner, Henry N., jun. (Div. Nucl. Med., Johns Hopkins Med. Inst., Baltimore, Md., U.S.A.). *J. Nucl. Med.*, 1977, **18** (4), 394–398.—Toadfish serum (TFS) has several advantages over other proteins for use as binder in a competitive-binding assay for cyanocobalamin. It is unaffected by changes in pH in the range 5·6 to 9·4 or by the addition of human-serum albumin. Prolonged incubation with charcoal does not disrupt the TFS - cyanocobalamin bond, and the addition of albumin as source of protein in the standard tubes is unnecessary. The binding capacity of TFS does not increase significantly with increasing concn. of cyanocobalamin. For 44 sera, the cyanocobalamin content determined by protein binding with TFS was higher than the value obtained by the conventional microbiological method with *Lactobacillus leichmannii*. CHEM. ABSTR.

1D148. **Radio-immunoassay for serum transcobalamin II.** Morelli, Thomas A.; Savage, C. Richard, jun.; Begley, James A.; and Hall, Charles A. (Hematol. Res. Lab., V.A. Hosp., Med. Coll., Union Univ., Albany, N.Y. 12208, U.S.A.). *J. Lab. Clin. Med.*, 1977, **89** (3), 645–652.—The method described, involving use of the stable labelled complex transcobalamin II (**I**) - [^{57}Co]cyanocobalamin, is simpler to carry out than that of Schneider *et al.* (*J. Clin. Invest.*, 1976, **57**, 27). The preparation of reagents and the assay procedure are described. The method is responsive to both saturated and unsaturated **I**, and whole serum containing a known amount of **I** can be used for calibration. Neither cyanocobalamin nor cyanocobalamin-binding protein interferes. The coeff. of variation (10 results) for a sample containing 1440 pg of **I** was 11%. D. W. S.

1D149. **Assay of enzymic activity by electrochemical detection of reduced coenzyme.** Wallace, Thomas C.; and Coughlin, Robert W. (Dept. Chem. Eng., Lehigh Univ., Bethlehem, Pa. 18015, U.S.A.). *Anal. Biochem.*, 1977, **80** (1), 133–144.—The method involved use of a standard H-cell modified to permit vigorous and uniform magnetic stirring. The anode vol. was 5 to 8 ml, and the anode materials were platinum mesh, high-density graphite, vitreous carbon and porous stainless steel. A potential of +70 V (*vs.* the s.c.e.) was applied to the buffer soln. (0·1M-potassium phosphate, 2mM-NAD^{+} and 0·02M-Li DL-lactate) (pH 8·1), and the cell current was allowed to stabilise (5 to 7 min). The baseline current was recorded, then a sample of the enzyme preparation was added, and the resulting current *vs.* time response was recorded graphically. The method could be used for aq. soln. of lactate, alcohol and malate dehydrogenases, and the reproducibility was within ±10%. R. A. H.

1D150. **Rapid and simple determination of aldose reductase and sorbitol dehydrogenase [L-iditol dehydrogenase].** Matkovics, B.; Koncz, C.; and Szabo, L. (Biol. Isotope Lab., Inst. Biochem., 'A. J.' Univ., Szeged, Hungary). *Microchem. J.*, 1977, **22** (1), 65–69.—These two enzymes (**I** and **II**, respectively) were determined separately in tissue (*e.g.*, liver) homogenates. The method for **I** involved measurement of initial and final concn. of glucose by the *o*-toluidine method (Hyvarinen and Nikkila, *Anal. Abstr.*, 1962, **9**, 4350). **II** was determined by the decrease in concn. of added NADH measured at 340 nm, a calibration graph being rectilinear for 0·1 to 0·4 μmol of NADH. T. G. A.

1D151. **Quantitative assay of hydroxymethylglutaryl-CoA reductase: comparison of a thin-layer chromatographic assay with a rapid chloroform extraction assay.** Ackerman, Margaret E.; Redd, William L.; Tormanen, Calvin D.; Hardgrave, Joel E.; and Scallen, Terence J. (Dept. Biochem., Sch. Med., Univ. New Mexico, Albuquerque, N. Mex. 87131, U.S.A.). *J. Lipid Res.*, 1977, **18** (3), 408–413.—The t.l.c. method described, which is accurate and sensitive, but time-consuming, involves use of plastic strips (9·5 cm × 2 cm) pre-coated with Silica gel G impregnated with ammonium carbonate. After development with $CHCl_3$ - methanol (25:1), the strip is cut into 14 sections and each is assayed for the radioactivity of [4-^{14}C]mevalonolactone by scintillation counting. A wide and complete separation is achieved between 3-hydroxy-3-methylglutaric acid and mevalonolactone, and means are described for determining the radiochemical purity of the [4-^{14}C]mevalonolactone. The second method, which is as accurate as the t.l.c. method, but quicker, involves extraction with $CHCl_3$ of an incubation mixture saturated with solid equimolar KH_2PO_4 - K_2HPO_4 at pH 6·8 and scintillation counting of [4-^{14}C]mevalonolactone. Good correlation between the two methods was observed. C. B.

1D152. **Fluorimetric determination of aromatic aldehydes with naphthalene-1,2-diamine. III. New fluorimetric assay for human serum monoamine oxidase [amine oxidase (flavin-containing)].** Zaitsu, Kiyoshi; Nagai, Hideaki; Kohashi, Kazuya; and Ohkura, Yosuke (Fac. Pharm. Sci., Kyushu Univ., Fukuoka, Japan). *Chem. Pharm. Bull.*, 1977, **25** (5), 918–921.—A mixture of 0·4 ml of phosphate buffer soln. (pH 7·2) and 0·1 ml of serum, in a stoppered tube, is incubated at 37° for 5 min; 0·50 ml of 0·01M-benzylamine hydrochloride in the buffer soln. is added, and incubation is continued for 1 h. To determine the benzaldehyde formed, 2 ml of naphthalene-1,2-diamine sulphate soln. (45 μg ml^{-1}) is added, and the mixture is heated for 20 min on a boiling-water bath, cooled and treated with 2 ml of 10% NaOH soln. After ≃10 min, the fluorescence is measured at 390 nm (with excitation at 356 nm) and compared with that for standards. E. G. B.

1D153. **Lipoxygenase assay: comparison between spectrophotometric and polarographic methods. Assay by a new colorimetric method.** Drapron, R.;

and Nicolas, J. (Sta. Biophysicochem. Cereales, INRA au CERDI, Massy, France). *Riv. Ital. Sostanze Grasse*, 1977, **54** (5), 218–221.—A comparison is made between assays based on polarographic determination of O uptake and spectrophotometric determination of the increase in absorbance due to formation of conjugated double bonds. These procedures are considered to be suitable as reference methods, but a colorimetric method is recommended for routine control. The method of Surrey (*Plant Physiol.*, 1964, **39**, 65) is used for the enzyme reaction; at specified intervals, samples of the reaction mixture are withdrawn and added to ethanolic indigo carmine and $CuCl_2$ catalyst, and the soln. are incubated at 75° for 20 min, then cooled, and the increases in absorbance at 615 nm are measured; portions of the soln. are used for the measurement of conjugated double bonds at 234 nm. A rectilinear relationship between the two parameters indicated a good correlation between the bleaching of indigo carmine and the formation of double bonds. R. F. S.

1D154. **Improved radiochemical assay for benzopyrene 3-monooxygenase [flavoprotein-linked monooxygenase].** Nesnow, Stephen; Fahl, William E.; and Jefcoate, Colin R. (Dept. Human Oncol., Univ. Wisconsin Med. Sch., Madison, Wis. 53706, U.S.A.). *Anal. Biochem.*, 1977, **80** (1), 258–266.—The method was based on that of Depierre *et al.* (*Anal. Abstr.*, 1975, **29**, 4D158), and the microsomes were prepared by the method of van der Hoeven *et al.* (*J. Biol. Chem.*, 1974, **249**, 6302). The reaction mixture, containing microsomal protein, NADP (1 μmol), glucose 6-phosphate (4·5 μmol), glucose-6-phosphate dehydrogenase (0·36 i.u.), $MgCl_2$ (3 μmol), potassium phosphate buffer of pH 7·5 (50 μmol) and [^{3}H]benzo[*a*]pyrene (60 nmol; 1 μCi), was incubated at 37° for 15 min. The reaction was stopped by addition of 0·5M-NaOH in aq. 80% ethanol (1 ml), and the soln. was extracted (by vortex mixer) with hexane (3 ml). After centrifugation at 2500 r.p.m., trifluoroacetic acid (10 μl) was added to the hexane phase, which was subjected to vortex mixing and centrifugation, then a portion (300 μl) of the supernatant soln. was neutralised with 0·5M-HCl, and the radioactivity was measured in a scintillation counter. The results were comparable with those of h.p.l.c. R. A. H.

1D155. **Simplified titrimetric semi-micro determination of triacylglycerol lipase in serum.** Appel, W.; and Scholz, H.-G. (Zentrallab. u. Med. Klin., St. Vincentius-Krankenhaus, Karlsruhe, W. Germany). *J. Clin. Chem. Clin. Biochem.*, 1977, **15** (6), 339–343 (in German).—The method of Rick (*Ibid.*, 1969, **7**, 530) has been modified, without loss of specificity, for routine assay of 0·2-ml samples. The sample is mixed with 2 ml of 75mM-Na glycocholate and 20 μl of olive oil suspension ($\simeq$200 g l^{-1}) and the pH is quickly adjusted to 8·2 with 0·01M-NaOH. The soln. is then titrated with 0·01M-NaOH, with use of a glass electrode and a s.c.e. as reference. A rectilinear calibration graph was obtained for enzyme activity between 5 and 800 i.u. l^{-1}, the practical detection limit being 10 i.u. l^{-1}. The within-series coeff. of variation for 20 determinations of 68 i.u. l^{-1} of enzyme activity was 4·75%, but day-to-day variation was 18·8 to 28·5%. The method can also be applied to duodenal fluid and urine; it does not require an atmosphere of N, and 20 samples can be assayed in 1 to 1·5 h. T. H. N.

1D156. **New electro-analytical method for determination of [acetyl]cholinesterase activity.** Damokos, T.; Havas, J.; and Rado, A. *Orv. Tech.*, 1976, **14** (5), 147–149 (in Hungarian).—The method is based on determination of the amount of acetic acid liberated (per unit time) by action of the enzyme on, *e.g.*, acetylcholine. The determination is effected (within 3 to 5 min) by coulometric titration with NaOH in an apparatus equipped with means for accurately maintaining the pH of the soln. at 7·4 and for graphical recording of the results; the slope of the graph is directly proportional to the enzyme activity. Advantages of the method are discussed. HUNG. TECH. ABSTR.

1D157. **Effect of sucrose on spectrophotometric determination of cholinesterase activities.** Atterwill, C. K.; and Neal, M. J. (Dept. Pharmacol., Sch. Pharm., Brunswick Sq., London, W.C.1, England). *J. Pharm. Pharmacol.*, 1977, **29** (6), 386–387.—The apparent activities of acetylcholinesterase and cholinesterase, as measured by the method of Ellman *et al.* (*Biochem. Pharmacol.*, 1961, **7**, 88), are significantly increased in the presence of sucrose. For example, the rate of hydrolysis of 2·5mM-acetylthiocholine iodide at 37° is $\simeq$3 nmol min^{-1} in 0·05M-sucrose at pH 8·0. The effect is non-enzymic. A. R. R.

1D158. **Nuclease detection in sodium dodecyl sulphate - polyacrylamide gel electrophoresis.** Rosenthal, Allan L.; and Lacks, Sanford A. (Biol. Dept., Brookhaven Natl. Lab., Upton, N.Y. 11973, U.S.A.). *Anal. Biochem.*, 1977, **80** (1), 76–90.—The nuclease samples were separated by electrophoresis on such gels, which contained DNA or RNA, after which the gels were rinsed in H_2O and incubated overnight at 30° in 0·04M-Tris - HCl buffer (pH 7·6) containing 2mM-$MgCl_2$ and 0·02% of NaN_3. After washing, further buffer soln. (containing also 2mM-$CaCl_2$) was added, followed by ethidium bromide (1 μg ml^{-1}). The enzymes appeared as sharply defined dark bands on a fluorescent background of ethidium bromide bound to unhydrolysed nucleic acid. The method was used to identify the endo- and exo-nucleases of *Diplococcus pneumoniae* from crude cell extracts, and deoxyribonucleases and ribonucleases from extracts of *Escherichia coli*, *Bacillus subtilis*, HeLa cells and *Haemophilus influenzae*. R. A. H.

1D159. **Critical observations on use of phenolphthalein monophosphate as substrate for determining prostate acid phosphatase.** Gallati, H. (Diagn. Forschungsabt., F. Hoffmann-La Roche & Co. A.-G., Basel, Switzerland). *J. Clin. Chem. Clin. Biochem.*, 1977, **15** (6), 323–328 (in German).—The effect of proteins and alcohols on the intensity of colour in analysis for acid phosphatase with use of phenolphthalein monophosphate (**I**) was examined. Following incubation, the reaction mixture was adjusted to pH 10 and the absorbance at 546 nm, due to phenolphthalein, was measured. In the presence of albumin the absorbance was reduced and Beer's law was not obeyed, although this reduction in absorbance was less at higher pH. The absorbance of an albumin-free soln. was reduced by the addition of aliphatic alcohols (*e.g.*, butanol), but that of a soln. containing albumin was increased. In the incubation-pH range 4 to 8, albumin combined with **I**, reducing the true substrate concn. and shifting the optimum pH of the enzyme. In the absence of albumin, alcohols strongly activated the

enzyme, but, in the presence of albumin, this effect was much reduced. These interferences could lead to errors in the interpretation of the test results.
T. H. N.

1D160. **Electrophoresis of acid phosphohydrolase [phosphatase] isoenzymes on Cellogel.** Hodes, M. E.; Crisp, M.; and Gelb, E. (Dept. Med. Genet., Indiana Univ. Sch. Med., Indianapolis, Ind. 46202, U.S.A.). *Anal. Biochem.*, 1977, **80** (1), 239–248.—Cell extracts (prep. described) of leucocytes and of spleen were separated by electrophoresis on cellulose acetate strips (Cellogel RS; Kalex Scientific Co., N.Y.), and the isoenzymes of acid deoxyribonuclease and ribonuclease, 3′-phosphodiesterase and non-specific phosphodiesterase were located with use of zymograms prepared from filter-papers or agarose-coated glass slides containing the appropriate substrate. The method is rapid and reproducible and requires relatively small amounts of blood (up to 2 ml) or spleen tissue. R. A. H.

1D161. **The estimation of leucine aminopeptidase [aminopeptidase (cytosol)] produced by moulting nematodes with labelled leucinamide as substrate.** Rogers, W. P.; and Brooks, Frances (Dept. Entomol., Waite Agric. Res. Inst., Glen Osmond, S. Australia). *Microchem. J.*, 1977, **22** (2), 245–248.—The enzyme activity is measured by using L-[U-^{14}C]leucinamide (prep. described) as substrate; the liberated leucine is separated by high-voltage electrophoresis and determined by scintillation counting for ^{14}C. The sensitivity is improved by this method, which also simplifies assay of enzyme activity during the moulting of infective juveniles of *Haemonchus contortus* in borate - CO_2 medium at 38°, as no purification or concentration of the enzyme is needed. W. J. B.

1D162. **Clinical application of serum leucine aminopeptidase [aminopeptidase (cytosol)] and monoamine oxidase [amine oxidase (flavin-containing)] estimation by direct colorimetric determination of ammonia.** Ito, Susumi; Takaoka, Takeshi; Kishi, Seiichiro; Okuda, Hiromichi; and Fujii, Setsuro (Second Dept. Internal Med., Sch. Med., Tokushima Univ., Japan). *Tokushima J. Exp. Med.*, 1977, **24** (1–2), 33–40.—The NH_3 is liberated by the enzyme-catalysed reactions from leucinamide (for the aminopeptidase) and butylamine or benzylamine (for the oxidase). The reaction is stopped by adding acid Na_2WO_4 soln., and NH_4^+ in the resulting supernatant soln. are determined by a reaction involving formation of a coloured indophenol (Okuda and Fujii, *Saishin Igaku*, 1966, **12**, 622), with measurement at 630 nm. Results for the aminopeptidase agreed with those obtained by using a commercial kit. Results for normal and pathological sera are discussed. It was possible, by a chromatographic method, to separate the oxidase activity into two fractions, one of which was more responsive to butylamine than to benzylamine; butylamine is therefore preferred as substrate for this enzyme. B. H.

1D163. **Determination of adenosine deaminase activity using high-pressure liquid chromatography.** Uberti, Joseph; Lightbody, James J.; and Johnson, Robert M. (Wayne State Univ. Sch. Med., Gordon H. Scott Hall Basic Med. Sci., Detroit, Mich. 48201, U.S.A.). *Anal. Biochem.*, 1977, **80** (1), 1–8.—The lymphocyte fraction of blood was prepared as previously described (*J. Immunol.*, 1974, **112**, 890), and the lysed cell suspension (10 µl) was added to a soln. (1 ml) that was 50mM in imidazole (pH 7) and 200µM in adenosine. The mixture was incubated at 37° for 1 h, and the reaction was stopped by adding trichloroacetic acid soln. (1 g ml^{-1}) (50 µl); the ppt. was centrifuged off, and the excess of the acid was removed by extraction with ethyl ether. The supernatant soln. was subjected to h.p.l.c. on a column of SCX-10 (Reeve-Angel) with 2mM-$(NH_4)_3PO_4$ (pH 3·5) as eluent (30 ml h^{-1}), and the adenosine deaminase activity was determined by measurement of the absorbance (at 254 nm) of the eluate, which contained inosine and hypoxanthine formed in the enzyme reaction. As little as 0·008 i.u. of enzyme could be detected, and the method could be applied to erythrocytes and lymphocytes.
R. A. H.

See also Abstracts: **1A7**, Assays for steroids. **1B49**, **1B50**, Determination of Au in plant material. **1B61**, Determination of Sr in environmental samples rich in Ca. **1B173**, Determination of Se in bovine liver. **1C21**, Analysis of saccharides. **1C23**, Determination of D-xylose. **1C36**, Titration of cephalosporins. **1C52**, Chromatographic separation of drug metabolites. **1C73**, Identification of dyes in living tissue. **1C103**, Determination of deoxyribose in tissue. **1E10**, **1E11**, Analysis of vegetable drugs. **1F3**, Trace analysis for Cd, Pb, Hg and methylmercury: review. **1F5**, **1F6**, Determination of Pb in biological materials. **1F11**, Determination of Mn in tea. **1G1**, Determination of *N* in protein. **1G9**, Determination of B in plant material. **1G11**, Determination of P in plant material. **1G13**, Determination of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin in plant material. **1G18**, Determination of bromacil in murine tissue. **1G20**, Occurrence of ethylenethiourea in plants and animals. **1J3**, Grinding of plant material. **1J7**, Extraction of chemicals from tissue. **1J11**, Columns for separation of amino-acids. **1J12**, Column packing for separation of biochemicals. **1J19**, Chromatography of S-containing biological substances. **1J23**, Apparatus for electrophoresis. **1J24**, Gradient isoelectric focusing. **1J26**, T.l.c. of lipids. **1J27**, T.l.c. in enzyme assay. **1J38**, Detector for g.c. of drugs in body fluids. **1J65**, Furnace tubes for a.a.s. of Pb in blood. **1J67**, Spectral determination of Pb, Sn and Zn in plants. **1J72**, Cell for fluorescence analysis of biochemicals. **1J86**, Determination of riboflavine. **1J94**, M.s. of Ca in biological material. **1J100**, **1J101**, Liquid scintillation counting in biochemistry. **1J105**, Applications of ion-selective electrodes in biology and medicine. **1J113**, Measurement of purine bases.

E—PHARMACEUTICAL CHEMISTRY

1E1. **Identification and chemical classification of drugs based on relative response of a [rubidium-bead] nitrogen [- phosphorus] selective detector and flame ionisation detector in gas-chromatographic analysis.** Baker, John K. (Dept. Med. Chem., Sch. Pharm., Univ. Mississippi, University, Miss. 38677, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 906–908.—The samples are analysed on a glass column (183 cm × 2 mm) packed with 3% of OV-17 on Anachrom

ABS (110 to 120 mesh) and operated with He as carrier gas (27 ml min^{-1}); the column is temp.-programmed from 100° to 250° (at 4° min^{-1}) and then kept at 250° for 15 min. The drugs are characterised by their retention times relative to caffeine (the internal standard) or by the ratio of the detector response for the sample to that for the caffeine standard (response index). Response indices and relative retention times for 71 drugs containing N are listed. G. P. C.

1E2. **Use of programmable calculators in pharmaceutical analysis. II. Analysis of complex absorption spectra.** Kakac, B.; and Rejholec, V. (Res. Inst. Pharm. and Biochem., Prague, Czechoslovakia). *Cesk. Farm.*, 1977, **26** (4), 124–127 (in Czech).—In such spectra, overlapping maxima were approximated by means of Gauss or Lorentz functions, and the functional parameters were then used to calculate the desired values with a programmable calculator and graph plotter. The technique was applied to the u.v. spectra of a mixture of triamcinolone acetonide and chlorhexidine, to the i.r. spectra of the two isomers of clomiphene citrate and to the densitometry of thin-layer chromatograms. (For Part I see *Anal. Abstr.*, 1977, **33**, 6E1.) H. M. T.

1E3. **Applications of particle size analysis in the pharmaceutical industry.** Orr, N. A.; and Spence, J. (Wellcome Foundation Ltd., Dartford, Kent, England). *Analyst (London)*, 1977, **102** (1215), 466–472.—The significance of particle size distribution in a number of practical situations is discussed. Results obtained with a Coulter counter (model TA) are shown to be of value in monitoring a crystallisation process, in the control of grinding, in examination of disintegrated tablets, and in determination of amorphous insulin in an insulin - Zn suspension, the amorphous and crystalline forms being distinguishable on the basis of particle size distribution. R. W.

1E4. **Inorganic compounds in vegetable drugs. I. Chromatographic detection of several elements.** Gill, Stanislaw; and Luczkiewicz, Irena (Dept. Pharmacog., Med. Acad., Gdansk, Poland). *Farm. Pol.*, 1977, **33** (3), 145–149 (in Polish).—Calcium, Mo, Cu, Al, Fe, Ni, Co, Mn, Zn, Mg, Na, K, Li, P (as PO_4^{3-}) and S (as SO_4^{2-}) can be detected by t.l.c. on Silica gel G - MN 300 cellulose (3 : 7), with use of five mobile phases and nine detection reagents; the colours given by the elements with the reagents are tabulated, chromatograms are reproduced and procedures are detailed for detecting each element. The procedure permits detection of down to 1 μg of Al, Mn or Cu (with an alizarin reagent), Cu, Co or Zn (with a dithizone reagent), or Mn, Cu or Co (with a benzidine reagent); down to 3 μg of Na, K or Li can be detected (with a quercetin reagent). B. K.

1E5. **Rapid water determination in emulsion ointments.** Blum, [Mrs.] Ferenc; and Magyar, Maria. *Gyogyszereszet*, 1977, **21** (2), 57–60 (in Hungarian).—Water in such samples is determined with use of a butyrometer. The ointment is dissolved in $CHCl_3$ - benzene, any emulsion is broken by treatment with resorcinol or NaCl in the presence of HCl and polyoxyethylene glycol 400, and the amount of water is determined from the vol. of the aq. phase, corrected for the dilution caused by the reagents. The error is ± 1%. CHEM. ABSTR.

1E6. **Determination of residual moisture content in lyophilised products.** Polderman, J.; and Hooymans, P. M. (Dept. Pharm. Technol., State Univ., Leiden, Netherlands). *Pharm. Weekbl.*, 1977, **112** (23), 562–565.—Freeze-dried aminoacetic acid (glycine) was extracted with dioxan - benzene (1 : 9) containing pentane as internal standard. The extractant was added (by syringe) through the stopper of the container, which was agitated on a vortex-type mixer for 30 s. A sample of the liquid was then withdrawn by syringe and analysed by g.c. on a 2-m column of Porapak Q (80 to 100 mesh) operated at 170°, with H (60 ml min^{-1}) as carrier gas and katharometric detection. The H_2O was determined by comparison of the peak height with that for pentane. Tests on H_2O (0·39 mg) added to dry glycine (50 to 150 mg) gave good results. Factors affecting the lower limit of determination are discussed. B. H.

1E7. **[Detection and] determination of contamination of calomel by mercury(II) ions.** Pawlaczyk, Jan; and Szymkowiak, Maria (Dept. Inorg. and Anal. Chem., Med. Acad., Poznan, Poland). *Farm. Pol.*, 1977, **33** (4), 211–213 (in Polish).—The sample was extracted with H_2O, and the extract was submitted to two-dimensional t.l.c. on silica gel, with benzene and toluene as first and second mobile phases. The spot due to Hg^{2+} was located by comparison with standard Hg^{2+} subjected to the same t.l.c. procedure, and the Hg^{2+} were determined in an aq. extract of the spot colorimetrically with dithizone. From 3·46 to 25·68 μg of Hg^{2+} was found in 1-g samples of calomel. B. K.

1E8. **Tin(II) ion quantitation in pyrophosphate and polyphosphate radiopharmaceutical kits using differential pulse polarography.** McBride, Martin H. D.; George, Robert E.; Kessler, Wayne V.; and Shaw, Stanley M. (Bionucleonics Dept., Sch. Pharm. and Pharmacal Sci., Purdue Univ., West Lafayette, Ind. 47907, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 870–871.—To determine 0·1 to 1mM-Sn^{II} in the presence of Sn^{IV} in these soln., add the sample (1 ml) to degassed M-H_2SO_4 (9 ml) in the polarographic cell, and record the differential pulse scan from −0·3 to −1·0 V *vs.* the s.c.e.; use a 25-mV pulse, and scan at 5 mV s^{-1}. The peak height is a rectilinear function of the Sn^{II} concn. The coeff. of variation (4 determinations) was <7% for a pyrophosphate soln. and <17% for a polyphosphate soln. A. R. R.

1E9. **Spectrophotometric determination, with azo-dyes, of iron(II) or cobalt(II) ions in pharmaceuticals.** Domagalina, Eugenia; and Zareba, Stanislaw (Dept. Pharm. Chem., Med. Acad., Lublin, Poland). *Farm. Pol.*, 1977, **33** (4), 207–209 (in Polish).—[5-(2-Hydroxy-1-naphthylazo)-1,3,4-thiadiazol-2-yl]thioacetic acid forms a complex with Fe^{2+} at pH 7·2; Beer's law is obeyed for 0·12 to 2·35 μg ml^{-1} of Fe^{2+} and max. absorbance is at 766 nm. Two compounds, 1-(5-mercapto-1,3,4-thiadiazol-2-ylazo)-2-naphthol (**I**) and 4-(1,3,4-thiadiazol-2-ylazo)resorcinol (**II**), form complexes with Co^{2+} at pH 9·2; Beer's law is obeyed for 0·06 to 1·65 μg ml^{-1} of Co at 680 nm for **I** and for 0·06 to 1·47 μg ml^{-1} of Co at 560 nm for **II**. Analytical procedures are described for application to several pharmaceuticals containing Fe^{2+} or Co^{2+}. B. K.

1E10. **Physico-chemical method for the analysis of phytochemical preparations and raw materials of vegetable origin.** Georgievs'kii, V. P. (Chem.-Pharm. Inst., Kharkov, Ukr. SSR). *Farm. Zh. (Kiev)*, 1977, (1), 36–44 (in Ukrainian).—The literature published since 1970 on optical methods

for the identification and determination of biologically active substances, *e.g.*, alkaloids, is reviewed. (123 references.) A. S. W.

1E11. **Use of physico-chemical methods for studying the qualitative and quantitative composition of polysaccharides in raw materials and preparations of vegetable origin.** Dzyuba, N. P.; and Chushenko, V. M. (Chem.-Pharm. Inst., Kharkov, Ukr. SSR). *Farm. Zh.* (*Kiev*), 1977, (1), 56–59 (in Ukrainian).—Chromatographic methods are described for analysing the hydrolysate obtained by heating a polysaccharide preparation obtained from the leaves of *Plantago major* for 6 h at 100° with 2N-H_2SO_4. *Paper chromatography*—Butanol - pyridine - H_2O (6 : 4 : 3), ethyl acetate - acetic acid - H_2O (3 : 2 : 1) or butanol - acetic acid - H_2O (4 : 1 : 5) is used as developing solvent. The spots are visualised with acidic anilinium phthalate and alkaline $AgNO_3$ soln. *Gel-filtration chromatography*—A column of Bio-Gel previously treated with 0·05M-NaH_2PO_4 is used. The sample, dissolved in 1 ml of NaH_2PO_4 soln., is applied to the column, which is percolated with H_2O. *Ion-exchange chromatography*—A column of DEAE-cellulose ($H_2PO_4^-$ form) is used. The sample, dissolved in 1 ml of 0·05N-NaH_2PO_4, is applied to the column, and neutral polysaccharides are eluted with 0·05N-NaH_2PO_4, then acidic polysaccharides with 0·2N-KOH. *Gas chromatography*—The polysaccharides are converted into alditol acetates for analysis on a column packed with Gas-Chrom Q impregnated with 3% of HI-EFF 8BP and operated at 175°; a flame ionisation detector is used. A. S. W.

1E12. **Dinitrobenzenes (*ortho* and *meta*) and their derivatives as reagents in analysis of organic medicinal substances.** Yavors'kii, M. P. (L'vov Med. Inst., USSR). *Farm. Zh.* (*Kiev*), 1976, (5), 26–29 (in Ukrainian).—A review is presented of the use of *o*- and *m*-dinitrobenzene, 4,5-dinitroveratrole, 3,4- and 3,5-dinitrobenzoic acid, 1,3,5-trinitrobenzene, 3,5-dinitrobenzenesulphonic acid, bis-(2,4-dinitrophenyl) sulphone and picric acid in colorimetric analysis for various compounds that are reducing agents or contain an active methylene group. Compounds mentioned are—sugars (glucose, arabinose, lactose, maltose, fructose), uric acid, polyhydric phenols, tannin, ascorbic acid, adrenaline, isoniazid, cotarnine, physostigmine, streptomycin, gramicidin S, lobeline, apomorphine, khellin, hydrocodone, oxycodone, cortisone acetate, sex hormones, cardiac glycosides, dehydrocholic acid, 17-oxosteroids, citral, piperitone, alkaloids, and aldehydes and ketones. (45 references.) C. D. K.

1E13. **Use of high-pressure liquid chromatography for analysis of colouring materials.** Attina, M.; and Ciranni, G. (Ist. Chim. Farm. e Tossicol., Univ. Roma, Italy). *Farmaco, Ed. Prat.*, 1977, **32** (4), 186–191 (in Italian).—The colours E 102 [tartrazine (C. I. Food Yellow 4)], E 104 [quinoline yellow (C. I. Food Yellow 13)], E 122 [carmoisine (C. I. Acid Red 14)], E 110 [orange yellow S (C. I. Food Red 3)], E 123 [amaranth (C. I. Food Red 9)], E 126 [Ponceau 6R (C. I. Food Red 8)] and E 131 [Patent blue V (C. I. Acid Blue 3)], and extracts of E 102, of E 110 and of mixtures of E 110 and E 123 obtained from commercial pharmaceutical preparations (including multivitamin tablets and syrups), were rapidly separated and accurately determined by h.p.l.c. (at, *e.g.*, 122 atm) on Permaphase AAX in a medium of HCl - citrate buffer (pH 4 for E 104 and E 102; pH 5·1 for the other colours). The absorbance of the column percolate was measured at 240 nm (260 nm for E 102). The method was rapid, the only sample-preparation steps required being grinding, extraction with the h.p.l.c. solvent and filtration. E. C. A.

1E14. **Application of synthetic ion-exchange resins in analysis of pharmaceutical preparations. XIV. Determination of active ingredients in compound drugs.** Jarzebinski, Jerzy; and Posepna, Karin (Pharm. Anal. Lab., Med. Acad., Warsaw, Poland). *Farm. Pol.*, 1977, **33** (2), 77–80 (in Polish).—The method described in Part I (*Anal. Abstr.*, 1971, **21**, 2119) was used for determining 12 active ingredients, *e.g.*, amidopyrine, phenylbutazone, sulphathiazole, theobromine, papaverine hydrochloride and phenobarbitone, in 7 preparations. The results obtained by alkalimetric or complexometric (with edta soln.) titration of the H^+ or Zn^{2+} liberated from the ion-exchange column are compared with those obtained by various other methods. (For Part XIII see *Anal. Abstr.*, 1977, **32**, 3E15.) B. K.

1E15. **Determination of 2-aminoethyl dihydrogen phosphate by phosphorus-31 Fourier-transform nuclear magnetic resonance spectrometry.** O'Neill, Ian K.; and Pringuer, Martin A. (Lab. Govt. Chem., Stamford St., London, S.E.1, England). *Anal. Chem.*, 1977, **49** (4), 588–590.—The method was developed to determine this compound (**I**), of pharmaceutical interest, when dissolved in aq. 50% sucrose. Sodium pyrophosphate was used as internal standard because its sharp ^{31}P resonance is situated close to that of **I**, and $K_3Cr(CN)_6$ was used to overcome the pronounced relaxation effects caused by sucrose. The coeff. of variation was $\simeq$2% for duplicate assays of eight proprietary tonic soln. containing $\simeq$4% of **I**. G. P. C.

1E16. **Analytical evaluation of tannins in several crude drugs.** Malek, Bernard; and Zbierska, Janina (Inst. Med. Plants, Poznan, Poland). *Herba Pol.*, 1976, **22** (3–4), 243–246 (in Polish).—The contents of total tannins, catechol tannins and pyrogallol tannins were determined in tormentil rhizome, cowberry leaf, oak bark and silverweed (Herba Anserinae), and the ratios of catechol tannins to pyrogallol tannins were calculated. Aqueous extracts of the drugs were examined by t.l.c. on polyamide containing 5% of gypsum, with methanol - H_2O - formic acid (30 : 50 : 11) as mobile phase, and by electrophoresis on Whatman No. 3 paper for 6 h at 10·7 V cm^{-1} with borate buffer soln. of pH 9·2 as electrolyte. The results indicated similar compositions of tannins in the rhizome and the oak bark, and a preponderance of catechol tannins in cowberry leaf. Silverweed had the lowest contents of catechol tannins and of total tannins. B. K.

1E17. **New methods of separation, and behaviour of alkaloids on unmodified silica gel in very polar, neutral or acidic solvents. II.** Munier, R. L.; and Drapier, A. M. (C.N.R.S., Inst. Pasteur, Paris, France). *Chromatographia*, 1977, **10** (6), 290–297 (in French).—R_F values are presented for 12 alkaloids in mobile phases based on (usually) methanol, with chlorinated hydrocarbons, ethers, ethyl acetate, nitromethane, acetone, and acetic acid. Other mobile phases tested contained dioxan, acetonitrile and dimethyl sulphoxide. The mechanism of separation is discussed. (For Part I see *Anal. Abstr.*, 1977, **33**, 6E8.) M. J. Sa.

1E18. **Conductometric determination of several alkaloids in injections and tablets.** Jarzebinski, Jerzy; and Suchocki, Piotr (Pharm. Anal. Lab., Med. Acad., Warsaw, Poland). *Farm. Pol.*, 1977, **33** (3), 151–155 (in Polish).—The hydrochlorides of papaverine, pilocarpine, lobeline and ephedrine, and hyoscine hydrobromide, were determined in injections (*i*) by direct conductometric titration with 0·01N or 0·005N-NaOH in aq. ethanol medium, or (*ii*) by addition of 0·01N-NaOH and ethanol and conductometric titration with 0·01N-HCl. Method (*i*) could also be used for determining atropine and phenobarbitone (**I**) in Bellaphenal tablets, and **I** and theobromine in Theophenal tablets. Procedures are described for the determination of each component, and for the separation of **I** and alkaloids from tablets. B. K.

1E19. **Detection of homoeopathic drugs from solid vehicles. I.** Munshi, G. K.; Mudgal, V.; and Chanda, R. (Central Drugs Lab., Calcutta, India). *J. Inst. Chem.* (*India*), 1976, **48** (6), 297–298.—Commercial aconite, belladonna and nux vomica preparations were analysed by t.l.c., with benzene - ethyl acetate - diethylamine as mobile phase and Dragendorff reagent for detection. Several preparations lacked the active ingredients. CHEM. ABSTR.

1E20. **Quantitative determinations of codeine phosphate, guaiphenesin, pheniramine maleate, phenylpropanolamine hydrochloride and pyrilamine [mepyramine] maleate in an expectorant by high-pressure liquid chromatography.** Das Gupta, V.; and Ghanekar, A. G. (Coll. Pharm., Univ., Houston, Tex. 77004, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 895–897.—Codeine phosphate, guaiphenesin, Na benzoate, phenylpropanolamine hydrochloride, F D & C Yellow No. 5 (C. I. Acid Yellow 23) and the sum of pheniramine maleate (**I**) and mepyramine maleate (**II**) can be determined directly in a cough syrup after dilution with 0·05M-KH_2PO_4 soln. in aq. 13% methanol by h.p.l.c. on a μBondapak C_{18} column (30 cm × 4 mm) with the KH_2PO_4 soln. as mobile phase (2 ml min^{-1}) and detection at 254 nm. **I** is determined colorimetrically at 480 nm after reaction with CNBr. **II** is determined by difference, or spectrophotometrically at 315 nm after dilution with 0·1N-HCl; a small correction must be applied for interference from the dye. A. R. R.

1E21. **Determination of individual alkaloids in ergotamine, ergotoxine and ergocristine.** Kowalewska, Krystyna; and Speichert, Henryk (Inst. Med. Plants, Poznan, Poland). *Herba Pol.*, 1976, **22** (3–4), 275–280 (in Polish).—Various mixtures of ergot alkaloids were submitted to t.l.c., with colorimetric determination (Allport's reagent) of the individual components. The ergotamine and the ergotoxine alkaloids were separated on a 0·5-mm layer of Silica gel G, with acetone - benzene (2 : 1) as mobile phase, and the ergocristine alkaloids on a similar layer of diatomaceous earth G (Merck) impregnated with formamide - acetone - conc. aq. NH_3 (15 : 84 : 1), with heptane - ethyl acetate - dimethylformamide (300 : 250 : 1) as mobile phase. Results are presented for ergotamine and ergotaminine, ergotoxine and ergotoxinine, and ergocristine and ergocristinine. B. K.

1E22. **Thin-layer chromatography of some ergot alkaloids.** Sallam, L. A. R.; Naim, N.; and El-Refai, A. H. (Microbiol. Chem. Res. Lab., Natl. Res. Centre, Dokki, Cairo, Egypt). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 47–48.—R_F values are tabulated for 15 alkaloids of the clavine, lysergic acid amide, ergotamine and ergotoxine groups on Silica gel D (Riedel-de Haen) and Silica gel HF_{254} (Merck) plates with use of $CHCl_3$ - benzene - ethanol (40 : 10 : 3), $CHCl_3$ - methanol (18 : 1) and $CHCl_3$ - methanol - anhyd. acetic acid (900 : 50 : 1) as developing solvents, and some useful separations are indicated. The alkaloids were detected under 254-nm radiation and by spraying with 0·125% *p*-dimethylaminobenzaldehyde soln. in 50% H_2SO_4. Lysergic acid did not migrate in any of the systems cited. H. L. L.

1E23. **Extraction - photometric determination of tropane derivatives.** Bubon, N. T.; and Senov, P. L. (Med. Inst., Vitebsk, USSR). *Farmatsiya* (*Moscow*), 1977, **26** (3), 34–38 (in Russian).—Dissolve 1 mg of sample [atropine sulphate, tropacine (**I**), homatropine hydrobromide or hyoscine hydrobromide] in 100 ml of H_2O, and to 1 ml of the soln. add 3 ml of mM-bromocresol purple and 2 ml of buffer soln. (pH 3 for **I**, 4 for the others). Extract the coloured 1 : 1 product into $CHCl_3$ (2 × 5 ml); dilute the combined extracts to 10 ml with $CHCl_3$ and measure the absorbance at the absorption max. (405 to 410 nm). Obtain the alkaloid content from a calibration graph. Hydrolysis products of the alkaloids do not interfere unless present in threefold amounts. The relative error of the method does not exceed ±1%. C. D. K.

1E24. **Stability of drugs. III. Thin-layer and gas-chromatographic investigation of scopolamine [hyoscine] hydrobromide and its degradation products in eye-drops.** Goeber, B.; Timm, Ulrike; and Doehnert, H. (Sekt. Chem., Ber. Pharm., Humboldt Univ., Berlin, E. Germany). *Zentralbl. Pharm. Pharmakother. Laboratoriumsdiagn.*, 1977, **116** (1), 13–20 (in German).—Hyoscine and its decomposition products scopoline, apohyoscine and tropic acid can be identified by t.l.c. on Kieselgel G; R_F values in five solvent systems are reported. For quantitative work, 1 to 2 ml of sample is evaporated to dryness at 50° in a rotary evaporator and 0·5 ml of 0·65% atropine soln. in dimethylformamide is added as internal standard. To 0·1 ml of the resulting soln. is added 0·05 ml of bis(trimethylsilyl)acetamide, with heating at 80° for 2 min, and a 3-μl portion is subjected to g.l.c., with flame ionisation detection, on a packed column (10% of SE-30) operated either isothermally at 240° or with temp.-programming from 100° to 240° at 8° min^{-1}. G. E. F.

1E25. **Spectrophotometric determination of atropine methonitrate.** Zurawski, Piotr; and Ostrowska, Barbara (Inst. Med. Plants, Poznan, Poland). *Herba Pol.*, 1976, **22** (3–4), 213–221 (in Polish).—When atropine methonitrate (**I**) is treated with conc. H_2SO_4, the specific absorbance of the product is much higher than that of **I** in N-H_2SO_4 medium; this reaction has been used for the spectrophotometric determination of **I**. The sample (50 to 300 μg) was treated with 2 ml of conc. H_2SO_4 at 60° for 5 min, then the mixture was diluted with H_2O (8 ml) for spectrophotometry at 270 nm. Beer's law was obeyed in the range stated, and the mean error was <0·04%. Results agreed with those of titration with 0·05N-$HClO_4$ in non-aqueous medium. B. K.

1E26. **Spectrometric determination of ephedrine and methoxamine by hydroxylation.** Halmekoski, Jaakko; and Karki, Arja (Div. Pharm. Chem., Sch. Pharm., Univ., Helsinki, Finland). *Farm. Aikak.*, 1976, **85** (4), 125–128.—The drugs were determined

after monohydroxylation with H_2O_2 as oxidising agent, and with Fe^{3+} and catechol as catalysts, glucose being used as stabiliser when required. The products were coupled with 4-aminoantipyrine, and the absorbance was measured at 505 nm for ephedrine (**I**) and 490 nm for methoxamine (**II**). Calibration graphs were rectilinear for 0·1 to 0·8mM-**I** and 0·25 to 1·75mM-**II**. Tablet excipients did not interfere. C. B.

1E27. **Determination of berberine in crude drugs by fluorimetric densitometry.** Hashimoto, Yohei; Ando, Keiko; and Mizuno, Mizuo (Women's Coll. Pharm., Kobe, Japan). *Shoyakugaku Zasshi*, 1976, **30** (2), 127–131 (in Japanese).—Samples of, *e.g.*, *Coptis japonica*, *C. quinquefolia* or *Phellodendron amurense* were dried at 60° for 8 h, de-fatted with ether and extracted with methanol. The extracts were treated with 10% HCl to convert berberine into the hydrochloride, then subjected to t.l.c. on Kieselgel G, with butanol - acetic acid - H_2O (7 : 1 : 2) and cyclohexane - diethylamine (9 : 1) as developing solvents and determination by fluorimetric densitometry at 510 nm (with excitation at 353 nm). The contents of berberine ranged from 0·06 to 10·90%. CHEM. ABSTR.

1E28. **Application of the densitometric method to analysis of crude drugs. II. Determination of alkaloids in *Coptidis rhizoma* by thin-layer electrophoresis.** Noguchi, Mamoru; and Mochida, Kenshu (Natl. Inst. Hyg. Sci., Osaka, Japan). *Shoyakugaku Zasshi*, 1976, **30** (1), 47–51 (in Japanese).—Berberine and coptisine in *Coptidis rhizoma* are separated by constant-potential (900 V) electrophoresis on polyamide at 20°, with 5M-acetic acid as electrolyte. The alkaloids are determined by fluorescence densitometry at 550 nm, with excitation at 365 nm. Commercial samples contained 5·7 to 6·8% of berberine and 1·0 to 1·2% of coptisine. CHEM. ABSTR.

1E29. **Turbidimetric determination of berberine sulphate.** Popov, A. I.; Bazhina, L. M.; Oleshko, G. I.; and Kudymov, G. I. (Perm Pharm. Inst., USSR). *Farmatsiya* (*Moscow*), 1977, **26** (2), 78–79 (in Russian).—Dissolve 0·01 g of powdered drug or 0·3 g of ground tablets in H_2O, dilute to 200 ml, filter if necessary, and to 5 ml of filtrate add 5 ml of 0·01M-HNO_3, 1 ml of 5% Na_2SO_4 soln. and 5 ml of 0·01M-KI, and dilute to 20 ml. Place the soln. in a 5-cm spectrophotometer cell, and titrate with 0·25mM-$Bi(NO_3)_3$, with stirring at ≃650 r.p.m.; measure the absorbance at 620 nm after each addition of titrant until max. absorbance is reached. Determine the end-point from the intersection of the two rectilinear sections of the titration graph. For tablets (5 mg of active ingredient in each), the mean error was $<\pm 1{\cdot}9\%$. C. D. K.

1E30. **Ultra-violet spectrophotometric determination of aminophylline, amylobarbitone and ephedrine hydrochloride in an antiasthma capsule preparation.** De Fabrizio, Fabrizio [Saphar Laboratories (Pty) Ltd., Industria West, Johannesburg, S. Africa]. *J. Pharm. Sci.*, 1977, **66** (6), 811–813.—Prepare a suspension of the capsule contents, containing ≃65 mg of aminophylline (**I**) and ≃12·5 mg each of amylobarbitone (**II**) and ephedrine hydrochloride (**III**), in 50 ml of ethanol - acetic acid (1 : 1), centrifuge, and apply 10 ml of the supernatant liquid to a column (50 cm × 2 cm) packed with alginic acid (4 g) that has been pre-washed with 2N-HCl and ethanol - acetic acid (1 : 1). Wash the column with ethanol - acetic acid (1 : 1) at 1 ml min^{-1}, and collect 100 ml of eluate (soln. *A*), which contains **I** and **II**. Wash the column with H_2O (2 × 10 ml), then elute **III** with 0·1N-HCl at 2 ml min^{-1}; discard the first 7 ml of eluate, collect the next 100 ml, and determine **III** by spectrophotometry at 257 nm (4-cm cells). To determine **I** and **II**, adjust the pH of 2 ml of soln. *A* to 4, and that of a second 2-ml portion to 10 with 0·02N-NaOH, dilute each soln. to 50 ml with H_2O, and measure the absorbance of the soln. at pH 10 (with the soln. at pH 4 in the reference cell) at 285·5 nm (for **I**) and 240 nm (for **I** plus **II**) in 1-cm cells. Mean recoveries were >98·5%, and the standard deviations (10 determinations) were $<1{\cdot}11\%$. A. R. R.

1E31. **Potassium tetraiodobismuthate in absorptiometric analysis of euphylline [aminophylline].** Kristaleva, L. B.; and Kalugina, Z. G. (Perm Pharm. Inst., USSR). *Farmatsiya* (*Moscow*), 1977, **26** (2), 76–77 (in Russian).—Dissolve 0·3 g of sample in 100 ml of H_2O, filter, and to 1 ml of filtrate add 2 ml of $KBiI_4$ soln. [heat 1 g of $Bi(NO_3)_3$ with 10 ml of conc. H_2SO_4 for 20 min, dilute to 100 ml, and mix equal vol. of this soln. and 5% KI soln.]. Set aside for 1 h, filter off the pptd. complex, wash it with N-H_2SO_4 until the washings are colourless, then dissolve it in acetone, dilute to 25 ml with acetone, and measure the absorbance of the orange - yellow soln. *vs.* acetone. The relative error is $<\pm 5{\cdot}5\%$. C. D. K.

1E32. **Use of thin-layer chromatography in analysis for erysimin.** Mikhno, V. V.; and Kramarenko, V. P. (Zaporozhe Med. Inst., USSR). *Farm. Zh.* (*Kiev*), 1976, (5), 47–50 (in Ukrainian).—*Detection of erysimin* (**I**)—Apply the test soln. to a layer of silica gel, and develop by the ascending method with benzene - ethanol (7 : 3) or $CHCl_3$ - methanol (17 : 3); dry the layer, and, after 15 min, spray it with a 0·075% soln. of bis-(2,4-dinitrophenyl) sulphone (**II**) in ethanolic 1·5N-NaOH, and identify **I** from the blue spot (R_F ≃0·64 and 0·53 in the respective systems). *Determination of* **I**—Mark out the zone, obtained as described above, with a planimeter needle, transcribe it to tracing paper and then to graph paper, and measure its area; find the content of **I** from a calibration graph of spot area *vs.* amount of **I**, or of square root of the area *vs.* log. amount of **I**. The graphs are rectilinear for 10 to 70 μg of **I**, and the mean error is ±3·2% and ±2·35%, respectively. Alternatively, extract the **I** zone from an unsprayed plate with 2 ml of 96% ethanol for 15 min, centrifuge for 10 min at 3000 r.p.m., and dilute the supernatant liquid to 2 ml with ethanol. To 1 ml of this soln. add 1·25 ml of 0·075% **II** soln. in 96% ethanol and 0·25 ml of 0·15N-KOH, and, after 4 min, measure the absorbance with use of a 5-mm cell and a red filter. The mean error is ±3·2%. C. D. K.

1E33. **Identification of cannabinoidic acids by thin-layer chromatography of their methyl esters.** Krejci, Z.; and Hanus, L. (Med. Fac., Palacky Univ., Olomouc, Czechoslovakia). *Acta Univ. Palacki. Olomuc., Fac. Med.*, 1976, **76**, 145–152.—Pure cannabidiolic acid and Δ^9-tetrahydrocannabinolic acid were more readily separated after methylation with diazomethane than as the free acids; the separation method was t.l.c., with hexane - dioxan - methanol (7 : 2 : 1) as mobile phase. The same procedure was used in separating components in the acidic fraction of two varieties of plant, except that

the mobile phase was hexane - ethyl acetate (4:1). The method offers a rapid and simple means for identification of cannabinoidic acids.

CHEM. ABSTR.

1E34. **Comparative determinations of essential oils in drugs according to the European and German pharmacopoeias.** Mechler, E.; and Kovar, K.-A. (Pharm. Inst., Univ. Tuebingen, W. Germany). *Dtsch. Apoth.-Ztg.*, 1977, **117** (26), 1019–1023 (in German).—Various steam-distillation techniques for determining essential oils are summarised, and the types of apparatus are illustrated. A tabular comparison is presented of the conditions recommended in the edited pharmacopoeias for use in analysing 10 vegetable drugs, and results obtained by the methods are compared statistically. For 5 of the 10 drugs included in the Ph. Eur. III and in the second supplement to the D.A.B. 7, there was no significant difference in the yields of essential oil. Better precision obtained by using the D.A.B. 7 method is attributed to the narrow divisions of the graduated tube. G. E. F.

1E35. **Analysis of essential oils by combined liquid - solid and gas - liquid chromatography. II. Monoterpenes in essential seed oil of *Anethum graveolens* L.** Scheffer, J. J. C.; Koedam, A.; and Baerheim Svendsen, A. (Dept. Pharmacog., Gorlaeus Lab., State Univ., Leiden, Netherlands). *Medd. Nor. Farm. Selsk.*, 1977, **39** (3), 161–187.—Oil from dill seed is separated into hydrocarbon and oxygenated-terpene fractions by column chromatography on Silica gel 60, with pentane and a gradient (2·5 to 50%) of ethyl ether in pentane as respective eluents. The resulting fractions are analysed by g.l.c. at 45°, 60° and 80° with three stationary phases (PEG 20M, 3,3′-oxydipropionitrile and SF-96), each supported on Chromosorb W-AW (60 to 80 mesh), in copper columns (8 m × 1·5 mm) with N as carrier gas and flame ionisation detection. The identities of the compounds producing the chromatographic peaks are discussed. (For Part I see *Anal. Abstr.*, 1977, **33**, 1E22.) B. H.

1E36. **Quantitative separation of cineole and β-phellandrene in eucalyptus leaf extracts.** Goodwin, C. L.; and Squillace, A. E. (U.S. Dept. Agric., Forest Serv., Southeastern Forest Exp. Sta., Olustree, Fla. 32072). *Phytochemistry*, 1976, **15** (11), 1771–1773.—Pentane extracts were concentrated before fractionation on silica gel (100 to 200 mesh), β-phellandrene being eluted with light petroleum and cineole with ethyl ether - light petroleum (1:9). The eluates were then analysed by g.l.c. on a copper column packed with Carbowax 20M on Chromosorb W (60 to 80 mesh), with flame ionisation detection and *p*-cymene as internal standard. C. B.

1E37. **Spectrophotometric determination of some volatile oils. II. Differential method for estimation of cinnamaldehyde, cuminaldehyde and carvone.** Salam, Nabil Abdel; Hadi, Mahmoud Abdel; and Hamid, Yehia Abdel (Fac. Pharm., Alexandria Univ., Egypt). *Acta Pharm. Jugosl.*, 1977, **27** (2), 81–86.—The shift in the wavelength of max. absorption of aromatic aldehydes and ketones following the semicarbazone condensation reaction was used as the basis for a differential spectrophotometric determination of these compounds in ethanolic soln.; measured values of change in absorbance per 1-nm shift for the test soln. were compared with those for a reference soln. containing the same compound in known concn. The results for cinnamaldehyde and cuminaldehyde in cassia, cinnamon, and cumin oils, and for carvone in caraway and dill oils, are compared with those obtained by the normal spectrophotometric method, demonstrating improved accuracy and freedom from interference. B. D. H.

1E38. **Assessment of thyme and its pharmaceutical preparations. I.** van Os, F. H. L.; and Elema, E. T. (Lab. Farmacog. Galen. Farm., Rijksuniv., Groningen, Netherlands). *Pharm. Weekbl.*, 1977, **112** (23), 557–562 (in Dutch).—Procedures are described for determining phenols and essential oil in thyme and its preparations. Phenols are determined by t.l.c. of an ethanol extract of the sample on Silica gel GF_{254}, with thymol and ursolic acid as reference compounds and $CHCl_3$ - methanol (19:1) as mobile phase. After spraying with anisaldehyde soln., thymol gives a red band ($R_F \simeq 0{\cdot}6$), and ursolic acid a violet one ($R_F \simeq 0{\cdot}3$). Other zones on the chromatogram are discussed. Essential oil is determined by a distillation method. The samples examined included *Thymus vulgaris* L., *T. zygis* L. and *T. pulegioides* L. B. H.

1E39. **Determination of chamazulene and prochamazulenes in essential oils and crude drugs from yarrow (*Achillea* spp.; Compositae). II. New colorimetric method of high sensitivity for determination of chamazulene in the essential oils.** Verzar (nee Petri), Gizella; and Cuong, Bhanh N. (Inst. Pharmacog., Semmelweis Med. Univ., Budapest, Hungary). *Acta Pharm. Hung.*, 1977, **47** (3), 134–141.—An investigation of the reaction of chamazulene (**I**) with 4-dimethylaminobenzaldehyde, as in the determination of **I** (*cf.* Part I, *Anal. Abstr.*, 1977, **33**, 3E45), is reported. The reaction product has an intense blue colour (absorption max. 642 nm) in basic soln. and a yellow colour in acidic soln., and can be used as an acid - base indicator. The reaction is applied to the determination of **I** in the essential oil by a method based on that used in Part I. The colour is stabilised by the addition of ethanol to the soln. after the reaction, and the reproducibility of the method is improved. The calibration graph is rectilinear for up to 3 mg l^{-1} of **I**. R. F. S.

1E40. **Determination of bornyl acetate in processing products of *Abies sibirica*.** Morozova, S. S.; Katyuzhanskaya, A. N.; Pekhov, A. V.; Koshevoi, E. P.; and Mikhailova, N. S. (Krasnodar Sci.-Res. Inst. Brew. Ind., RSFSR). *Khim. Prir. Soedin.*, 1976, (3), 303–306 (in Russian).—The method is based on the reaction of complex esters with alkaline hydroxylamine to form hydroxamic acids, which react with Fe^{III} to give a pink - yellow colour. *Procedure*—The essential oil (≃0·5 g) is dissolved in 25 ml of ethanol, and the soln. is passed through an alumina column. (A correction is subsequently made for the ≃11% loss on the column.) To 5 ml of the eluate are added 0·36 ml of 20% hydroxylammonium chloride soln. and 3·3 ml of 0·5N-NaOH. The soln. is set aside for 16 min, then 0·28 ml of 5N-HCl and 1·7 ml of 6% $FeCl_3$ soln. are added, and the absorbance is measured after 11 min at 400 to 450 nm. M. R. M.

1E41. **Practical method for identifying 'tonin' (peach kernel) and 'kyonin' (apricot kernel) with a urine-sugar-analysis paper (Tes-Tape).** Nagoshi, Kiro; and Nakano, Kimiko (Fac. Pharm. Sci., Univ. Tokushima, Japan). *Shoyakugaku Zasshi*, 1976, **30** (1), 42–46 (in Japanese).—The Chinese herbal medicines peach kernel and apricot kernel can

be distinguished by their amygdalin contents. The kernels were extracted with 10 vol. of H_2O, and the extracts, containing amygdalin, were hydrolysed to give glucose. The contents of glucose were measured with a conventional test paper. Apricot kernel yielded more glucose than did peach kernel.
CHEM. ABSTR.

1E42. **Gas - liquid chromatography of methyl esters of natural penicillins.** Messchaert, B.; Adriaens, P.; and Eyssen, H. (The Rega Inst., Univ. Leuven, Belgium). *J. Chromatogr.*, 1977, **136** (1), 162–164.—Penicillins were extracted into ethyl ether from aq. soln. at pH 2 and esterified with diazomethane at 0° to 2°. The ether soln. was evaporated to dryness, and the residue was re-dissolved in acetone and subjected to g.l.c. on a glass column (150 cm × 4 mm) packed with 3% of OV-1 or OV-17 on Gas-Chrom Q (100 to 120 mesh) and operated for 10 min at 150° or 180°, respectively, then temp.-programmed at 4° min^{-1} for 10 min, with N as carrier gas (60 ml min^{-1}).
M. J. SL.

1E43. **Determination of gentamicin-complex components in fermentation broth by *in situ* fluorimetric measurements of 4-chloro-7-nitrobenzofurazan derivatives.** Kabasakalian, Peter; Kalliney, Sami; and Magatti, Anita W. (Dev. Div., Schering Corp., Bloomfield, N.J. 07003, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 953–955.—The sample of broth was adjusted to pH 2 with H_2SO_4 and centrifuged, the supernatant liquid was adjusted to pH 12 with NaOH, and, after dilution, the soln. was subjected to t.l.c. (Wilson *et al.*, *Anal. Abstr.*, 1973, **25**, 2564). The plates were then dried in air, immersed in methanolic 4-chloro-7-nitrobenzofurazan (0·25 mg ml^{-1}) and subjected to a development with methanol; the spots were scanned fluorimetrically at 530 nm (excitation at 420 nm). The method was 800 times more sensitive than that based on ninhydrin.
G. P. C.

1E44. **Separation of bleomycins by high-pressure liquid chromatography.** Williams, C. R.; Gifford, L. A.; and Scanlon, B. (Dept. Chem., Univ. Technol., Loughborough, England). *Anal. Lett.*, 1977, **10** (5), 407–413.—Bleomycins were separated by h.p.l.c. on a column (30 cm × 0·4 cm) of μPorasil, with freshly prepared ammonium formate - methanol (1 : 1) as eluent (2 ml min^{-1}), and were monitored in the eluate at 254 nm. The u.v.-absorbing components of the eluate were freeze-dried and subjected to t.l.c. on silica gel with 10% ammonium formate - methanol (1 : 1) as developing solvent; individual bleomycins were located on the chromatogram under 254-nm radiation and were identified from their R_F values. This method, which was more rapid than conventional h.p.l.c. or radio-immunoassay methods, could also be used to separate Cu^{II} - bleomycin complexes.
I. R. H. C.

1E45. **Gas - liquid chromatographic determination of lasalocid and its bromo-analogue as their silyl derivatives.** Manius, Gerald; and Viswanathan, V. (Anal. Res. Lab., Quality Control Dept., Hoffmann-La Roche Inc., Nutley, N.J. 07110, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 769–772.—Mix the sample (*e.g.*, pellets, pre-mixes, mycelial cake, injection soln. or powder), containing ≃50 mg of lasalocid (**I**) or its bromo-analogue, with *NO*-bis(trimethylsilyl)acetamide, *N*-trimethylsilylimidazole and chlorotrimethylsilane (3 : 3 : 2) (1 ml), and set aside in a closed vessel for 30 min. Dilute the soln. to 10 ml with tetrahydrofuran, and submit 5 μl to g.l.c. at 260° in a stainless-steel column (61 cm × 3·2 mm o.d.) packed with 3% of methyl silicone fluid on acid-washed silanised diatomite, with N as carrier gas (60 ml min^{-1}) and flame ionisation detection. Measure the peak areas. The results are in good agreement with those obtained by titration or by microbiological assay; the coeff. of variation (6 determinations) is 1·41%. Meglumine and the major product of thermal degradation of **I** do not interfere.
A. R. R.

1E46. **Analytical studies on active constituents in crude drugs. III. High-speed liquid-chromatographic determination of ecdysterone and inokosterone in Achyranthes radix.** Ogawa, Shuntaro; Yoshida, Akiyoshi; and Kato, Reiko (Rohto Pharmaceutical Co. Ltd., Ikunoku, Osaka, Japan). *Chem. Pharm. Bull.*, 1977, **25** (5), 904–908.—The powdered root (10 g) is extracted with 200 ml of methanol for 5 h in a Soxhlet apparatus and the extract is evaporated to dryness under reduced pressure. The residue is leached with 25 ml of methanol, any insol. material is centrifuged off, and 5 μl is then directly injected into a liquid chromatograph equipped with a column (2 m × 2·1 mm) of Permaphase ETH at 50°; the mobile phase is ethanol - hexane (1 : 9), and detection is at 254 nm. Inokosterone can be resolved into its isomers (thought to be C_{25} epimers) on a column of Permaphase ODS by gradient elution with H_2O - methanol. (For Part II see *Anal. Abstr.*, 1977, **33**, 1E18.)
E. G. B.

1E47. **Determination of rutin in presence of ascorbic acid.** Sivitskaya, O. K.; Rubinskaya, V. G.; Kuleshova M. I.; and Guseva, L. N. (All-Union Sci.-Res. Inst. Pharm., Moscow, USSR). *Farmatsiya* (*Moscow*), 1977, **26** (2), 81–82 (in Russian).—Heat 0·02 g of the preparation with 15 ml of 96% ethanol on a water bath, cool, dilute to 25 ml with the ethanol, and filter if necessary (*e.g.*, if Ca gluconate is present). Dilute an aliquot to 5 ml with ethanol, add 4·5 ml of H_2O and 0·5 ml of 0·1N-NaOH, and, after 15 min, measure the absorbance of the soln. at 415 nm against 96% ethanol - H_2O (1 : 1). Calculate the rutin content from the absorbance of a standard soln. (0·5 ml of ethanol containing 0·1 mg of rutin, 0·5 ml of fresh 0·1% ascorbic acid soln., 4·5 ml of 96% ethanol, 4 ml of H_2O and 0·5 ml of 0·1N-NaOH). There is no interference from nicotinic acid, pyridoxine hydrochloride, thiamine bromide, glucose, sucrose or Ca gluconate. Recovery of rutin ranges from 88 to 96·5%.
C. D. K.

1E48. **Barbituric acid derivatives. XXIV. Determination of enantiomeric purity of chiral barbiturates with lanthanoid shift reagents.** Knabe, Joachim; and Gradmann, Volker (Fachr. Pharm. Chem., Univ. Saarland, Saarbruecken, W. Germany). *Arch. Pharm.* (*Weinheim, Ger.*), 1977, **310** (6), 468–473 (in German).—It is demonstrated that, with use of a medium of $CDCl_3$ containing, *e.g.*, tris-[3-heptafluorobutyryl-(+)-camphorato]europium^{III}, the n.m.r. spectrum of hexobarbitone shows separate signals attributable to the (*S*)- and (*R*)-configurations of the methyl groups at the 1- and 5-positions; down to 4% of the racemate in the (*S*)-(+)-form can be detected. The cited shift reagent has also been used in detection of the racemate in (*S*)-(+)-5-butyl-1-methyl-5-phenylbarbituric and (*S*)-(+)-5-(cyclohex-1-enyl)-1-ethyl-5-methylbarbituric acids.
R. L. J.

1E49. **Automatic amperometric micro-titration of barbiturates with mercury(II) at the glassy-carbon electrode. Comparison of one- and two-phase**

titrations. Coenegracht, P. M. J.; Bult, A.; and Metting, H. J. (Lab. Pharm. and Anal. Chem., Groningen, Netherlands). *Fresenius' Z. Anal. Chem.*, 1977, **284** (4), 273–281.—Optimum conditions have been established for titrating six barbiturates with 4 or 40mM-$Hg(NO_3)_2$ in 0·01M-HNO_3 by use of an indicator electrode made from a vitreous-carbon rod and a silver - AgCl reference electrode (satd. KNO_3 bridge). Two single-phase titration procedures, in media of 0·5M-KNO_3 and 0·1M-Na acetate, respectively, were compared with a two-phase titration procedure, and all three methods were compared with a macro-titration (Dutch pharmacopoeial procedure) as reference method. For determination of barbiturates in the range 10 to 100μM, the two-phase method was preferred; 50 μg of barbiturate could be titrated with a precision and accuracy of $\simeq \pm 2\%$. For the two-phase method, the test soln. was added to 7 ml of 0·1M-Na acetate and 3 ml of organic solvent [$CHCl_3$ for titration of amylobarbitone sodium or pentobarbitone; $CHCl_3$ - benzyl alcohol (4:1) for phenobarbitone, phenobarbitone sodium, barbitone or barbitone sodium]. Titrant was delivered from an automatic burette controlled by a stepping motor driven by a signal from the titration cell. C. K. L.

1E50. **Application of orthogonal functions to spectrophotometric determination of phenytoin and phenobarbitone in pharmaceutical preparations.** Amer, M. M.; Ahmad, A. K. S.; and Hassan, S. M. (Anal. Chem. Dept., Fac. Pharm., Univ. Cairo, Egypt). *J. Pharm. Pharmacol.*, 1977, **29** (6), 355–357.—Extract phenytoin (**I**) and phenobarbitone (**II**) from the mixture or capsules (containing $\simeq$100 mg of **I**) with 0·1N-NaOH (50 ml). Dilute to 100 ml with 0·05M-borate buffer soln. of pH 10, and dilute 1 ml of this soln. to 100 ml with the same buffer soln. Measure the absorbance at 6-nm intervals from 226 to 256 nm and at 240 nm. Calculate the **II** concn. by means of the quadratic polynomial p_2 at the six equally spaced wavelengths, and use it to calculate the contribution of **II** to the total absorbance at 240 nm and hence (from the difference) the **I** concn. The mean recoveries and coeff. of variation (9 determinations) were—for **I**, 100·0 ±1·3%; for **II**, 100·0 ± 1·4%. A. R. R.

1E51. **Determination of aspirin in Citramon tablets.** Tiraspol'skaya, S. G.; and Terenik, A. P. (Pyatigorsk Pharm. Inst., USSR). *Farmatsiya* (*Moscow*), 1977, **26** (2), 68–69 (in Russian).—Shake 0·5 g of powdered tablets with 10 ml of $CHCl_3$ for 10 min, filter, wash the residue with $CHCl_3$ (2 × 5 ml), remove $CHCl_3$ from the combined filtrate and washings by evaporation on a water bath, dissolve the residue in 20 ml of ethanol, and titrate with 0·1N-NaOH (phenolphthalein as indicator). C. D. K.

1E52. **Quantitative determinations of antipyrine [phenazone] and benzocaine in ear-drops by high-pressure liquid chromatography.** Das Gupta, V.; and Sachanandani, S. (Coll. Pharm., Univ., Houston, Tex. 77004, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 897–898.—Dilute the sample (0·5 ml), containing 5·4% of phenazone (**I**) and 1·4% of benzocaine (**II**), to 100 ml with H_2O, and submit 10 μl to h.p.l.c. on a μBondapak C_{18} column (30 cm × 4 mm) operated at room temp., with 0·02M-KH_2PO_4 soln. in aq. 40% methanol as mobile phase (1·6 ml min^{-1}) and detection at 254 nm. Oxyquinoline sulphate, used as a preservative, does not interfere and the assay appears to indicate stability. The coeff. of variation (4 determinations) was 0·68% for **I** and 1·18% for **II**. A. R. R.

1E53. **Spot reaction for the detection of dicaine [amethocaine] in medicinal preparations.** Pesakhovich, L. V.; and Beketov, B. N. (Med. Inst., Tyumen, RSFSR). *Farm. Zh.* (*Kiev*), 1977, (1), 90–91 (in Ukrainian).—Spot an aq. soln. of the sample on to filter-paper and dry. Then moisten the paper with M-HCl and expose it to Br vapour until a yellow colour develops. Add one drop of 2·5% $NaNO_2$ soln., followed by one drop of 8% NaOH soln. containing 0·25% of 8-hydroxyquinoline sulphate. A red spot indicates the presence of amethocaine; the detection limit is 0·25 μg. The method is rapid and specific, but resorcinol, nitrofurazone and compounds containing a primary aromatic amino-group interfere. A. S. W.

1E54. **Use of ion-exchange chromatography for monitoring quality of sulphocamphocaine.** Grizodub, A. I.; and Kazarinov, N. A. (Nauchno-Issled. Khim. Farm. Inst., Kharkov, USSR). *Khim.-Farm. Zh.*, 1977, **11** (3), 134–136 (in Russian).—Camphorsulphonic acid, formed during the degradation of sulphocamphocaine preparations, was determined by adsorption on KU-2-8 cationite followed by absorbance measurement at 287 nm. Procaine (base), similarly formed, was determined by adsorption on anionite AV-17-8, with spectrophotometric measurement at 292 nm. CHEM. ABSTR.

1E55. **Spectrophotometric determination of procaine hydrochloride.** Luquez de Mucciarelli, Sara I.; and Lucas de Arellano, M. A. (Cat. Ensayo y Valor. Med., Univ. Nac., San Luis, Argentina). *Rev. Asoc. Bioquim. Argent.*, 1976, **41** (226), 179–182 (in Spanish).—A portion of the test soln. (filtered if necessary) is adjusted to 3 ml with H_2O and 2 ml of a 0·5% soln. of 2,5-dimethoxytetrahydrofuran in anhyd. acetic acid is added. After heating at 100° for 20 min, the mixture is cooled in ice and 5 ml of acetic acid and 2 ml of a 2% soln. of *p*-dimethylaminobenzaldehyde in acetic acid - HCl (17:3) are added. After 2 min in a boiling-water bath and cooling in ice, 10 ml of H_2O is added. The deep-pink colour, formed in 20 min and stable for 40 min, is measured at 550 nm. Beer's law is obeyed for procaine hydrochloride concn. between 0·02 and 0·06 mg ml^{-1}. J. K. C.

1E56. **Stability of aqueous solutions of pirbuterol.** Bansal, P. C.; and Monkhouse, D. C. (Central Res., Pfizer Inc., Groton, Conn. 06340, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 819–823.—To determine pirbuterol in aq. soln., submit the sample (5 μl, containing 2·5 to 25 μg) to t.l.c. on a 0·25-mm layer of Silica gel GF, with acetic acid - H_2O - ethyl methyl ketone (1:1:6) as solvent. Dry the chromatogram, and use a spectrodensitometer to measure the integrated reflectance (at 282 nm) of the spot at $R_F \simeq 0{\cdot}43$. A. R. R.

1E57. **Gas- and high-pressure liquid-chromatographic properties of some 4-nitrobenzamides of amphetamines and related (arylalkyl)amines.** Clark, C. Randall; Teague, J. Dwayne; Wells, Martha M.; and Ellis, Joel H. (Sch. Pharm., Auburn Univ., Ala. 36830, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 912–915.—The derivatives are prepared by reaction of the amines with 4-nitrobenzoyl chloride in tetrahydrofuran and M-NaOH at 65°. The mixture can be analysed directly, or after purifica-

tion by $CHCl_3$ extraction, by h.p.l.c. (at ambient temp.) on a column (30 cm × 0·25 in.) of μBondapak C_{18}, with H_2O - acetonitrile (13 : 7) as the eluent and 254-nm detection, or by g.l.c. (at 255°) on a column (5 ft. × 2 mm) packed with, *e.g.*, 3% of OV-17 on Chromosorb (80 to 100 mesh) and operated with CH_4 as carrier gas (20 ml min^{-1}) and chemical-ionisation m.s. detection. G. P. C.

1E58. **Stability of drugs. V. Analysis and stability of tropicamide.** Timm, Ulrike; Goeber, B.; Doehnert, H.; and Pfeifer, S. (Sekt. Chem., Ber. Pharm., Humboldt Univ., Berlin, E. Germany). *Pharmazie*, 1977, **32** (6), 331–335 (in German).—Impurities (*e.g.*, degradation products) associated with tropicamide (**I**) include apotropicamide (**II**) and *N*-ethyl-*N*-(4-pyridylmethyl)phenylacetamide (**III**) [which are formed in an alkaline soln. of **I** (*e.g.*, in an aq. diagnostic soln.)], tropic acid and *N*-(4-pyridylmethyl)ethylamine (**IV**). With the object of identifying **I** and assessing its purity, **II**, **III** and **I** picrate were prepared (details are given); t.l.c. data for all the compounds and spectroscopic (u.v., i.r. and n.m.r.) data for **I**, **II** and **III** are reported. From the information presented, it is concluded that the m.p. range for **I** and its picrate, the t.l.c. behaviour and d.t.a. values offer the most suitable criteria for purity assessment. **I**, **II** and **III** can also be determined by g.l.c. (details are given). From a study of reaction kinetics, it is deduced that cryst. **I**, and neutral or acid soln. thereof, are stable for at least 20 years at 20°, and the stability of **I** in alkaline medium is >1000 times that of atropine. F. R. B.

1E59. **Identification and determination of sulphonamides. I. Infra-red spectra.** Kalova, Helena; Slechtova, Rostislava; Socha, Jaromir; and Bekarek, Vojtech (Prirodovek. Fak., Palacky Univ., Olomouc, Czechoslovakia). *Acta Univ. Palacki. Olomuc., Fac. Rerum Nat.*, 1976, **49** (Chem. 15), 131–142.—The i.r. spectra (4000 to 400 cm^{-1}) of 40 sulphonamides have been measured in liquid paraffin mulls and in soln. in acetonitrile and chloromethane. Use of the spectra for identification purposes is suggested. CHEM. ABSTR.

1E60. **Investigation of Schiff bases of medicinal sulphonamides. II. Polarographic determination of some sulphonamides by Schiff-base reaction.** Lehtinen, M.; and Halmekoski, Jaakko (Div. Pharm. Chem., Univ., Helsinki, Finland). *Farm. Aikak.*, 1976, **85** (2), 35–43.—Sulphamethizole, sulphamethoxypyridazine and sulphafurazole were each treated with 5-bromosalicylaldehyde, and the resulting Schiff base was dissolved in a 0·05M soln. of tetraethylammonium iodide in dimethylformamide and determined polarographically. The three sulphonamides (down to 25, 22 and 20 mg, respectively) could be determined in tablets by this method, with coeff. of variation from ±1·41 to ±2·67% and without interference from excipients. The results obtained were comparable with those of spectrophotometric and colorimetric methods.
CHEM. ABSTR.

1E61. **Use of platinum - graphite biamperometric indication for determination of sulphonamides.** Prasad, B. B.; Khandelwal, G. D.; and Singh, T. B. (Dept. Chem., Fac. Sci., Banaras Hindu Univ., Varanasi, India). *Acta Pol. Pharm.*, 1977, **34** (2), 177–180.—Sulphanilamide, sulphapyridine, sulphaguanidine (**I**), sulphathiazole (**II**), sulphamerazine (**III**) and sulphadiazine are determined as 0·1mM to mM soln. in aq. HCl - KBr medium by titration with 4mM to 0·05M-$KBrO_3$, with use of an L-shaped platinum micro-electrode and a graphite electrode impregnated with paraffin wax. Results are tabulated for **I**, **II** and **III**; the errors for these range from −0·23% (≃0·28 mM-**III**) to −0·67% (≃0·88 mM-**I**).
F. R. B.

1E62. **Photometric determination of thiacetazone.** Laipanov, A. Kh. (Kursk Med. Inst., USSR). *Pharm. Zh.* (*Kiev*), 1976, (5), 55–57 (in Ukrainian).—*Detection of thiacetazone* (**I**)—Dissolve 5 to 10 mg of sample in 5 ml of dimethylformamide (**II**), and add 5 ml of 0·5% sodium amminepentacyanoferrate (**III**) soln. **I** gives an emerald-green colour (detectable minimum, 30 μg ml^{-1}). *Determination of* **I**—Dissolve the sample (≡0·05 g of **I**) in **II**, dilute to 50 ml with **II**, filter if necessary, and to 3 ml of the soln. add 2 ml of **II** and 5 ml of 0·5% **III** soln.; dilute to 25 ml with H_2O, and, after 30 min, measure the absorbance at 597 nm. Beer's law is obeyed for 0·05 to 0·5 mg ml^{-1} of **I**. The mean error is ±0·97% for the substance and within ±1·40% for tablets.
C. D. K.

1E63. **Use of rhodanine derivatives for identification and photometric determination of solutizone {sodium [4-(thiocarbamoylmethylhydrazonomethyl)-anilino]methanesulphonate}.** Svinchuk, V. S.; Kramarenko, V. P.; and Turkevich, B. M. (Med. Inst., Lvov, Ukr. SSR). *Farm. Zh.* (*Kiev*), 1977, (1), 66–67 (in Ukrainian).—Add 0·2 ml of 10% HCl to 1 ml of H_2O containing ≃0·1 mg of this drug (used in treating tuberculosis). Gently heat to the b.p. for 5 min, cool, add 0·2 ml of a 10% $NaNO_2$ soln. and, after 5 min, add 0·7 ml of a 0·1% aq. soln. of 2-(4-oxo-2-thioxothiazolidin-3-yl)glutaric acid. After 3 min add 1·5 ml of 10% NaOH soln., and dilute the soln. to 10 ml with H_2O. Measure the absorbance at 490 nm against a reagent blank. Calculate the solutizone concn. from a calibration graph prepared with standard preparations. Beer's law is valid from 0·005 to 0·13 mg of solutizone in the 10 ml of reaction mixture. A. S. W.

1E64. **Sensitive assay procedure for ethambutol hydrochloride via charge-transfer-complex formation.** Tan, Henry S. I.; Gerlach, Eric D.; and Dimattio, Anthony S. (Coll. Pharm., Univ. Cincinnati Med. Center, Cincinnati, Ohio 45267, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 766–769.—Shake the sample, containing ≃50 mg of ethambutol hydrochloride (**I**), with 8% NaOH soln. (3 ml), extract with $CHCl_3$ (5 × 15 ml), dry the combined extracts with Na_2SO_4, filter, and dilute to 100 ml with $CHCl_3$; dilute 5 ml to 50 ml with $CHCl_3$. Mix 1 ml of this soln. with 0·8mM-iodine in $CHCl_3$ (5 ml), dilute to 10 ml with $CHCl_3$, set aside in the dark for 1 h, and measure the absorbance at 293 nm against a reagent blank. Recoveries (5 determinations) ranged from 96·2% to 101·2%; the coeff. of variation (13 determinations) was 1·12%. A. R. R.

1E65. **Determination of Medamin [methyl benzimidazol-2-ylcarbamate] by non-aqueous titration.** Kharitonova, S. I. (E. I. Martsinovskii Inst. Med. Parasitol. and Tropical Med., Moscow, USSR). *Farmatsiya* (*Moscow*), 1977, **26** (2), 77 (in Russian).—Dissolve 0·15 g of the anthelmintic in 10 ml of anhyd. acetic acid (**I**), add 3 ml of acetic anhydride and 3 drops of 0·1% crystal violet soln. in **I**, and titrate with 0·1N-$HClO_4$ in **I**. The relative error is <±0·12%. C. D. K.

1E66. **Study of the stability of pyrimidine-series cytostatics, ftorafur and fluorouracil injections.** Tomankova, Hana; and Zyka, Jaroslav (Inst. for Control of Drugs, Prague, Czechoslovakia). *Microchem. J.*, 1977, **22** (1), 70–84.—In an investigation on the effects of temp., u.v. radiation and pH on the stability of ftorafur (**I**) and fluorouracil (**II**) injections, methods were developed for the separate determination of **I**, **II** and urea (a degradation product). The methods involved t.l.c. on Silufol UV 254, Lucefol and Lucefol Quick (Kavalier), with use of any one of eight solvent systems, and spectrophotometry at 272 nm (for **I**), 267 nm (**II**) and (after complex formation with Ehrlich reagent) 414 nm (urea). Results are reported for 0·4 and 1·0% soln. of **I** and **II**. B. T. S.

1E67. **Identification of corticosteroids by toluene-*p*-sulphonic acid on thin-layer chromatograms.** Madan, Vijay K.; and Sharma, S. C. (Central Dugs Lab., Calcutta, India). *J. Inst. Chem. (India)*, 1977, **49** (1), 62–64.—The colours given by cortisone acetate, dexamethasone, hydrocortisone acetate, prednisolone and prednisone after t.l.c. on Silica gel G and spraying with toluene-*p*-sulphonic acid soln., with observation in daylight and u.v. radiation, are reported as a function of heating time at 110°. In daylight, all the corticosteroids gave distinctly different colours; these did not change markedly until after 25 min of heating. The colours observed in u.v. radiation changed rapidly during heating; these changes could be used as additional identification tests. CHEM. ABSTR.

1E68. **Impurities in labelled stilboestrol: identification of pseudostilboestrol.** Tennent, D. M.; Kouba, R. F.; Ray, W. H.; VandenHeuvel, W. J. A.; and Wolf, F. J. (Rhodia Inc., Hess and Clark Div., Ashland, Ohio 44805, U.S.A.). *Science*, 1976, **194** (4269), 1059–1060.—Tests on stilboestrol (**I**) labelled with ^{14}C and intended for implantation in cattle showed that the preparations were impure. On two-dimensional t.l.c. on Silica gel G, with CH_2Cl_2 as the first mobile phase and benzene - ethyl ether (19 : 1) as the second, with autoradiography of the chromatogram, nine zones containing ^{14}C were obtained. Two were the *cis*- and *trans*-forms of **I**, and 2·3% of the ^{14}C remained at the origin; of the other zones, one contained 2·1% of the ^{14}C, and this was subsequently identified, by 70-eV m.s. of the material alone and after treatment with silylating reagent, as pseudostilboestrol (α-ethyl-α′-ethylidenebibenzyl-4,4′-diol). B. H.

1E69. **Hexane - acetonitrile partition systems applied to assay of corticosteroid ointments.** Fairbrother, J. E. (Squibb International Dev. Lab., Moreton, Merseyside, England). *Methodol. Dev. Biochem.*, 1976, **5**, 141–144.—Use of such a system for clean-up in analysis for, *e.g.*, triamcinolone acetonide or halcinonide in samples containing liquid paraffin and/or glycols or polysorbates is discussed. A soln. of the sample in hexane is shaken with acetonitrile, the acetonitrile layer is evaporated to dryness, and a soln. of the residue in $CHCl_3$ is analysed for the steroid by spectrophotometry after reaction with isoniazid or tetrazolium blue. For samples containing glycols (which interfere in the reaction with tetrazolium blue), preliminary partition between $CHCl_3$ and H_2O is necessary; the $CHCl_3$ soln. is then evaporated, and a soln. of the residue in hexane is treated as described above. The technique is also a useful preliminary to h.p.l.c. or g.l.c. B. H.

1E70. **Separation and quantitative determination of 2,4-disulphamoyl-5-trifluoromethylaniline in hydroflumethiazide using high-pressure liquid chromatography.** Owen, B. J.; and Wilkie, S. V. (Linson Ltd., Swords, Dublin, Eire). *J. Pharm. Sci.*, 1977, **66** (6), 877–878.—Prepare a methanolic soln. containing 0·04% of hydroflumethiazide and 0·04% of sulphathiazole (as internal standard), and submit 3 μl to h.p.l.c. at 35° on a stainless-steel column (50 cm × 2·6 mm) packed with Perkin-Elmer ODS-SIL-X-I, with aq. 5% methanol as mobile phase (1·5 ml min^{-1}) and detection at 254 nm. Measure the peak heights. For 10 determinations on a sample containing ≃0·97% of the amine, the coeff. of variation was 2·9%. A. R. R.

1E71. **Reactions of pentacyanoferrate(II) ions. V. Spectrophotometric study and determination of some antidotes against organophosphorus compounds.** Smeric, Zlata; Hankonyi, Vladimara; and Karas-Gasparec, Vinka (Dept. Chem. and Biochem., Fac. Med., Univ. Zagreb, Yugoslavia). *Acta Pharm. Jugosl.*, 1977, **27** (2), 97–104.—The reactions of amminepentacyanoferrate^{II} and pentacyanonitrosylferrate^{II} ions with three pharmaceutical quaternary pyridinium aldoximes {pralidoxime chloride, 1,1′-trimethylenebis-(4-hydroxyiminomethylpyridinium) dibromide and 4-(hydroxyiminomethyl)-1-[3-(4-methylmorpholino)propyl]pyridinium dichloride} are described. The complexes formed were used for spectrophotometric determinations at wavelengths in the range 525 to 590 nm, the effect of pH and NaOH concn. on the absorbance being investigated, and the precision under optimum conditions being calculated for 25 to 250-μg amounts of each compound. The method was also used for the determination of pralidoxime chloride in tablets (0·5 g per tablet) and for injection soln. (1 g per ampoule), the coeff. of variation of 0·46 to 1·4% being less than those obtained with the conventional u.v. spectrophotometric method. B. D. H.

1E72. **Ultra-violet absorption spectra of phenolic compounds. III. Hydroxybenzoic acid derivatives.** Milch, Gyorgy; and Aninger (nee Deak), K. (Chinoin Chemical Works, Budapest, Hungary). *Acta Pharm. Hung.*, 1977, **47** (3), 125–133 (in Hungarian).—Ultra-violet absorption spectra of hydroxybenzoic acid preservatives in various solvents (buffers having various values of pH, ethanol, cyclohexane, ethyl ether and dioxan) are discussed. Methods are given for the determination of such compounds in Hungarian pharmaceutical preparations, including the Na 2-(4-hydroxybenzoyl)benzoate content of Libexin (prenoxdiazin hydrochloride) antitussive agent. V. B.

1E73. **Simple gas - liquid chromatographic determination of ethylene oxide [oxiran] and its reaction products in drugs and formulations.** Hartman, P. A.; and Bowman, P. B. (Control Anal. Res. Dev. Lab., The Upjohn Co., Kalamazoo, Mich. 49001, U.S.A.). *J. Pharm. Sci.*, 1977, **66** (6), 789–792.—For H_2O-insoluble drugs, mix the sample (1 g) with H_2O (4 ml) for 5 min, centrifuge, and use the supernatant liquid. For ointments, dissolve the sample (3 g) in hexane (6 ml), extract by mixing for 5 min with H_2O (3 ml), centrifuge, and use the aq. phase. Analyse aq. samples directly. To determine oxiran (**I**), submit 6 μl of the aq. soln. or extract to g.c. at 110° on a glass column (180 cm × 3 mm) containing Porapak R (80 to 100 mesh), with He as carrier gas (60 ml min^{-1}) and flame ionisation detection; measure the peak heights. To determine 2-chloro-

ethanol (**II**) and ethanediol (**III**), submit a second 6-μl portion to g.l.c. under similar conditions, but with a column containing 15% of Carbowax 400 on Gas-Chrom Q (80 to 100 mesh). The limits of detection were 2 p.p.m. for **I** and **II** and 5 p.p.m. for **III**, and the mean recoveries were all >95%. Methanol interfered with the determination of **I** at 110°, but was separated from **I** at 70°. Benzyl alcohol interfered with the determination of **III**, but was separated from **III** on a Porapak column at 170°. A. R. R.

See also Abstracts: **1A7**, Assays for steroids. **1C8**, Determination of N in cephalexin. **1C10**, Determination of N in sulphathiazole. **1C36**, Titration of cephalosporins. **1C52**, Chromatographic separation of drugs. **1D28** to **1D90**, Determination of drugs and their metabolites in biological materials. **1D123**, Determination of dehydroepiandrosterone. **1D128**, Determination of sapogenins. **1F31**, Determination of capsaicinoids. **1F32**, Analysis of asafoetida. **1G16**, Determination of oestradiol. **1H16**, Determination of organomercury preservatives. **1J38**, Detector for g.c. of drugs and essential oils. **1J105**, Applications of ion-selective electrodes in medicine.

F—FOOD

1F1. **Hexanal as a measure of rancidity in low-fat foods.** Fritsch, C. W.; and Gale, J. A. (General Mills Inc., James Ford Bell Tech. Center, Plymouth Ave. North, Minneapolis, Minn. 55427, U.S.A.). *J. Am. Oil Chem. Soc.*, 1977, **54** (6), 225–228.—A 15-g sample (of, *e.g.*, breakfast cereal) is suspended in boiling water, and hexanal in the evolved vapour is determined by g.l.c. at 100° on an aluminium column (10 ft. × 0·25 in.) packed with 10% of OV-101 on acid-washed Chromosorb W (60 to 80 mesh), with He (40 ml min^{-1}) as carrier gas, flame ionisation detection and heptan-4-one as internal standard. The method is rapid and reproducibility is good. An increase in the concn. of hexanal above 5 p.p.m. produced the onset of rancid odour. The method was also applied to dehydrated potato, dried wheat gluten, defatted wheat germ and soya products. E. A.

1F2. **Comparing methods of analysis for selected trace elements in turkey muscle.** Zenoble, Oleane C.; Bowers, Jane A.; Seaman, Gregory; and Howe, Steven D. (Dept. Foods and Nutrit., Kansas State Univ., Manhattan, Kans. 66506, U.S.A.). *J. Food Sci.*, 1977, **42** (3), 847–848.—A comparison was made of a.a.s., n.a.a. and X-ray fluorescence (x.r.f.) for the determination of Zn, Fe, Rb and Cu in dried turkey breast. For Zn, a.a.s. and n.a.a. gave similar results, but x.r.f. values were significantly higher than a.a.s. values. For Rb, n.a.a. and x.r.f. gave similar results. Copper and Fe were not detectable by n.a.a., nor was Cu by x.r.f. In general, a.a.s. was the most precise method. R. F. S.

1F3. **Cadmium, lead, mercury and methylmercury compounds: review of methods of trace analysis and sampling with special reference to food.** Schuller, Pieter L.; and Egan, Harold (Rijks Inst. v. Volksgezondheit, Bilthoven, Netherlands). *FAO Rep.*, No. 92–5-1-00094-M-84, 1976. Pp. 97.—Analysis for each of these constituents is discussed under the general headings—sampling, destruction of organic matter, pre-concentration and separation, and quantification. The methods of determination considered include activation analysis, m.s., g.l.c., X-ray fluorescence, fluorimetry, colorimetry, atomic spectroscopy and electrometric techniques. (454 references.) B. H.

1F4. **Routine screening method for toxic metals in foods.** Allenby, P.; Robertson, J. W.; and Shenton, F. C. (North-East Regional Analysts Dept., County Hall, Durham, England). *J. Assoc. Public Anal.*, 1977, **15** (2), 61–68.—A method is described for determining Pb, Cu, Cd, Zn and As at high or low concn. The sample (5 to 10 g of dry material or 15 to 20 g of wet material), mixed with 20% H_2SO_4 (5 ml), is evaporated on a steam bath for 5 to 6 h, then carefully heated on a sand-bath or a low flame and finally ashed overnight in a furnace at 500°. The cold ash is heated on a steam bath for 15 min with 3N-HCl (10 ml), transferred to a flask, cooled, and diluted to 50 ml with water (soln. *A*). High levels of Pb, Cu, Cd and Zn are determined directly in soln. *A* by flame a.a.s.; low levels are similarly determined after extraction into diethylammonium diethyldithiocarbamate soln. in heptan-3-one; and As in soln. *A* is measured by the Gutzeit method. High concn. of Ca and Fe have no great effect on the procedures, and the method is widely applicable; detection limits (20-g sample) are <0·1 p.p.m. of Pb or Cu, 0·02 p.p.m. of Cd and 0·05 p.p.m. of Zn. Recoveries of Pb, Cu, Cd and Zn from 15 different foods ranged from 82 to 116%. E. C. A.

1F5. **Comparison of methods of determination of lead.** Truffert, L.; Favert, M.; and Le Gall, Y. (Lab. Cent. Prefecture Police, Paris, France). *Ann. Falsif. Expert. Chim.*, 1976, **69** (742), 563–570 (in French).—A comparison has been made between the results of two dithizone colorimetric methods [a general method (Truffert, *Ibid.*, 1972, **65**, 51) for use with foods and biological materials and a simplified method (Boudene *et al.*, *Igiena*, 1973, **22**, 37) for use with atmospheric samples] and of square-wave and pulse-polarographic methods. A statistical analysis of the results is presented. A comparison has also been made between the simplified dithizone method and flameless a.a.s. for the determination of atmospheric Pb. R. F. S.

1F6. **Determination of lead by atomic-absorption spectrophotometry in relation to toxicological research.** Oudart, N.; Guichard, C.; and Delage, C. *J. Eur. Toxicol.*, 1976, **9** (2), 69–73 (in French).—The possible use of a.a.s. for determining Pb in foodstuffs, water, industrial atmospheres and biological fluids has been studied. The principles of the technique (including sample preparation) are outlined, and typical results are discussed in terms of reproducibility, sensitivity and specificity. LEAD ABSTR.

1F7. **Application of physico-chemical methods to determination of lead in wine.** Petrino, P.; Cas, M.; and Estienne, J. (Univ. Technol., Marseille, France). *Ann. Falsif. Expert. Chim.*, 1976, **69** (737), 87–99 (in French).—The Pb was determined by a.a.s. after various sample treatments, and by polarography. For Pb contents <0·3 p.p.m., a.a.s. after chelation with ammonium pyrrolidine-1-carbodithioate and

extraction into isobutyl methyl ketone was preferred. For higher contents, dry-ashing with subsequent a.a.s., or polarography, was satisfactory.
CHEM. ABSTR.

1F8. **Elimination of sulphite interference in the spectrophotometric determination of nitrite.** Lew, R. B. (Amstar Corp., Spreckels Sugar Div., Woodland, Calif. 95695, U.S.A.). *Analyst* (*London*), 1977, **102** (1215), 476–479.—The determination of NO_2^- in sugar juices, by conventional colorimetric methods, is subject to interference from SO_3^{2-}; this interference can be eliminated by the use of formaldehyde. *Procedure*—To ≃20 ml of sample soln. (clarified, if necessary) containing 4 to 30 μg of NO_2^- add 2 ml of 5% formaldehyde soln., 5 ml of 0·2% sulphanilamide soln., and 2 ml of 6M-HCl, mixing after each addition. After 3 min add 1 ml of 0·2% *N*-(1-naphthyl)-ethylenediamine dihydrochloride, mix, dilute to 50 ml and after 2 min measure the absorbance at 540 nm.
R. W.

1F9. **Determination of arsenic in beverages and food by hydride-generation atomic-absorption spectroscopy.** Siemer, Darryl D.; Vitek, Richard K.; Koteel, Prabhakaran; and Houser, W. C. (Dept. Chem., Marquette Univ., Milwaukee, Wis. 53233, U.S.A.). *Anal. Lett.*, 1977, **10** (5), 357–369.—The sample (5 g for liquids; 0·5 g for anhyd. solids), mixed with 5 ml of satd. $Mg(NO_3)_2$ soln., is dry-ashed at 450°, the ash is dissolved in 1 ml of H_2O plus 4 ml of 9M-HCl, and the soln. is mixed with 0·5 ml of 0·1M-KI - 2% ascorbic acid and diluted to 10 ml with H_2O. An aliquot (0·5 or 1 ml) of this soln. is placed in a hydride generator followed by 1 ml each of 9M-HCl and aq. 1% $NaBH_4$, and the AsH_3 evolved is transferred to a H - O flame for analysis by a.a.s. The method can be used to determine concn. of As ranging from <10 p.p.b. to ≃9·3 p.p.m.
I. R. H. C.

1F10. **Comparative study of the determination of selenium in high-carbohydrate nutrient carriers using flameless atomic-absorption spectroscopy and fluorimetry.** Szydlowski, F. James (Ralston-Purina Co., St. Louis, Mo. 63188, U.S.A.). *At. Absorpt. Newsl.*, 1977, **16** (3), 60–63.—Selenium was determined in complex corn-oil-based matrices by a modification of the AOAC fluorimetric method and by the proposed flameless a.a.s. procedure. The samples (4-ml portions of infant foods and infant-food bases) were each digested with 10 ml of HNO_3 and then with 2 ml of $HClO_4$. The soln. was treated with M-HCl, then edta - hydroxylamine soln. was added and the pH was adjusted to 2·0 with aq. 7M-NH_3. Naphthalene-2,3-diamine (**I**) soln. was added, and the mixture was heated at 50° for 30 min before extracting the Se - **I** complex into 4 ml of dekalin. Selenium was determined in the organic soln. by fluorimetry with excitation at 375 nm and emission at 525 nm. Then 5 to 20-μl portions of the organic soln. were injected into the graphite furnace, followed by a 20-μl injection of 0·06% Cu soln. to prevent volatilisation losses, and Se was determined by a.a.s. at 196 nm with drying at 125° for 75 s, charring at 800° for 95 s and atomisation at 2700° for 13 s with an interrupted Ar flow. The samples contained between 2·6 and 18·7 ng ml^{-1} of Se. The two procedures gave results that showed good correlation, neither method giving results that were consistently higher or lower than those of the other. The average recovery of standard additions was 99% for the a.a.s. method and 104% for fluorimetry. It is concluded that, provided that precautions are taken to alleviate matrix interferences, flameless a.a.s. can give reliable results for samples with complex matrices.
C. K. L.

1F11. **Determination of manganese in tea by atomic-absorption spectrophotometry.** Chorazy, Wieslaw; Bliwert, Krystyna; and Swoszowska, Maria (Dept. Nutr. and Food Chem., Inst. Chem. and Med. Phys., Med. Acad., Sosnowiec, Poland). *Bromatol. Chem. Toksykol.*, 1976, **9** (3), 361–362 (in Polish).—The Mn (415 to 850 mg kg^{-1}) in 2 g of sample (dried for 1 h at 80°) is released by—(*i*) extraction with H_2O (50 ml) for 1 h at 100° followed by extraction with 10% HCl (24 ml) and wet oxidation of the extracts with H_2SO_4 - HNO_3 (1 : 10); (*ii*) wet oxidation for 2 h with H_2SO_4 - HNO_3 (1 : 10); or (*iii*) extraction for 15 min with hot 7·7% HCl for subsequent a.a.s. determination with use of the 279·5-nm line and an air - acetylene flame. The results obtained for the three variants are very similar, with no statistically significant differences.
P. B.

1F12. **Use of 5,6-diphenyl-3-(2-pyridyl)-1,2,4-triazine for determination of iron in wine.** Mandrou, B.; Fabre, H.; and Mattras, H. (Lab. Chim. Anal. Toxicol., Fac. Pharm., Montpellier, France). *Ann. Falsif. Expert. Chim.*, 1976, **69** (738), 219–228 (in French).—The method is the same as that of Schilt and Taylor (*Anal. Chem.*, 1970, **42**, 220), except that ethyl acetate is used as the extractant and H_2O_2 is used as an ashing aid. The results obtained are comparable with those of other colorimetric methods, the sensitivity is 1·7 times better than that of the standard 1,10-phenanthroline method, and Cu does not interfere. However, the method is not recommended (reasons for which are discussed).
CHEM. ABSTR.

1F13. **Determination of micro amounts of iron in fats and oils by atomic-absorption spectrophotometry.** Hirayama, Haruo; Shimoda, Zenya; Kobayashi, Akiyuki; Shige, Takeshi; Takahashi, Takeomi; Tsuji, Kenji; Nakasato, Satoshi; Higuchi, Masahiko; and Machida, Yoshiaki (Japan Oil Stuff Insp. Corp., Tokyo). *Yukagaku*, 1977, **26** (4), 216–220 (in Japanese).—The sample was decomposed by ashing with a filter-paper wick, the ash was dissolved in HCl, and the soln. was directly atomised in an air - acetylene flame for a.a.s. measurement at 248·3 nm. The method was evaluated with respect to recovery and reproducibility by carrying out collaborative studies on samples prepared by adding known amounts of Fe to soya-bean oil; the results obtained were in good agreement with theoretical values. The effect of the ashing technique on the recovery of Fe from representative samples is also discussed.
CHEM. ABSTR.

1F14. **Gas-chromatographic determination of low levels of phthalic acid esters in fish.** Ueta, Tadahiko; Yamazoe, Ritsuko; Mori, Kenichiro; Suzuki, Sukeji; Nishida, Shigekazu; and Totani, Tetsuya (Metrop. Res. Public Health, Tokyo, Japan). *Tokyo-toritsu Eisei Kenkyusho Kenkyu Nempo*, 1976, **27** (1), 100–105 (in Japanese).—Dibutyl phthalate (4 μg) and bis-(2-ethylhexyl) phthalate (4 μg) added to a 20-g sample of fish were extracted with ethyl ether - hexane (1 : 4), and the extract was concentrated and analysed by g.c. Recoveries of the added compounds were 70·2 and 87·9%, respectively, and the corresponding detection limits were 0·07 and 0·05 p.p.m.
CHEM. ABSTR.

1F15. **Migration behaviour and toxicology of methyltin stabilisers.** Uhde, W.-J.; and Woggon, H. (Zentralinst. Ernaehrung, Potsdam-Rehbruecke, E. Germany). *Nahrung*, 1977, **21** (5), 377–384 (in German).—Mono-, di- and tri-methyltin compounds used as stabilisers for poly(vinyl chloride) were separated by t.l.c. on Kieselgel G, with $CHCl_3$ - anhyd. acetic acid - ethyl ether (5:4:3) as mobile phase and ethanolic 0·2% quercetin as spray reagent. Monomethyltin compounds so isolated were determined as their quercetin complexes by spectrophotometry at 448 nm; dimethyltin compounds were determined by digestion of the excised zone with H_2SO_4 - HNO_3 followed by polarography of the Sn; and trimethyltin compounds were converted (by exposure of the plate to unfiltered u.v. radiation) into the dimethyltin derivatives for polarography. These methods were used in studying the migration of the stabilisers from poly(vinyl chloride) into food-simulant soln. (3% acetic acid, or 20 or 50% ethanol) and into the fat simulant HB 307. R. F. S.

1F16. **Investigations on the formation of mycotoxins and their quantitative determination. IX. Sterigmatocystin.** Engel, Guenter (Inst. Microbiol., Bundesanst. Milchforsch., Kiel, W. Germany). *J. Chromatogr.*, 1977, **136** (1), 182–185 (in German).—This mycotoxin was separated by t.l.c. on Kieselgel G 1500 plates (S. & S.) with benzene - ethanol (19:1) as developing solvent, and was detected by treatment with ethanolic $AlCl_3$ soln. followed by heating for 10 min at 105°; a derivative with a yellow fluorescence was formed by exposure to long-wave u.v. radiation. Sterigmatocystin was then determined by spectrodensitometry, either by measurement of u.v. remission at 245 nm (for amounts >0·05 μg at 360 nm) or by fluorimetry of the $AlCl_3$ derivative, with excitation at 360 nm and emission at 500 nm. The limits of detection were 0·01 μg and 0·02 μg, respectively, for the two techniques. R. F. S.

1F17. **Aflatoxin determination.** Anon. *Ind. Aliment. (Pinerolo, Italy)*, 1977, **16** (4), 85–86 (in Italian).—The food sample (50 g) was homogenised and extracted with aq. 85% acetone, the extract was filtered, and a portion of filtrate was shaken with $Fe(OH)_3$ gel. The gel was filtered off, the clear soln. was diluted, and the aflatoxins were extracted into $CHCl_3$; the extract was then evaporated, and the residue was dissolved in $CHCl_3$ - methanol (24:1). This soln. was passed through a micro-column containing ascending layers (in mm) of glass wool (2 to 3), sand (5 to 7), Florisil (5 to 7), sand (5 to 7), silica gel (15) and alumina (15), and the column was washed with $CHCl_3$ - methanol. The aflatoxins adsorbed by the Florisil were measured directly in a Fluorotoxin meter at 310 to 410 nm. The method was more sensitive and faster than the standard AOAC method. CHEM. ABSTR.

1F18. **Chemical analysis of mycotoxins. VII. Determination of aflatoxins by high-performance liquid chromatography.** Isohata, E.; Takeda, Y.; and Uchiyama, M. *Shokuhin Eiseigaku Zasshi*, 1977, **18** (1), 48–52 (in Japanese).—It was found that h.p.l.c. on silica gel (Iatrobeads 6RS-8010) gave good separation of aflatoxins B_1, B_2, G_1 and G_2 with CH_2Cl_2 - $CHCl_3$ - methanol (1200:800:3) or $CHCl_3$ - 2,2,4-trimethylpentane - isopropyl alcohol (150:50:1) as eluent, whereas a porous polymer (Iatrobeads 6CP-2010) could be used for the clear separation of B- and G-group aflatoxins from patulin and penicillic acid. The aflatoxins were detected at 360 nm, at which wavelength there was minimum interference from other constituents of the samples. (For Part VI see *Anal. Abstr.*, 1977, **32**, 6F12.) FOOD R. A. ABSTR.

1F19. **Comparison of two methods for analysis of aflatoxin M_1 in milk.** Bodine, A. B.; Davis, R. Wayne; Janzen, J. J.; and Crawford, F. M. (Dept. Dairy Sci., Univ., Clemson, S.C. 29631, U.S.A.). *J. Dairy Sci.*, 1977, **60** (3), 450–452.—Results obtained by the cellulose chromatography method of the AOAC (Pons *et al.*, *J. Assoc. Off. Anal. Chem.*, 1973, **56**, 1431) and a procedure with use of a silica gel mini-column and two-dimensional t.l.c. (Tuinstra and Bronsgeest, *Anal. Abstr.*, 1976, **30**, 4F9) are discussed. Results by the t.l.c. procedure agreed with those of the official procedure for aflatoxin M_1 concn. between 0·01 and 0·25 μg l^{-1}. Both methods gave recoveries >87% and there was no significant difference between variances. The t.l.c. procedure is less time-consuming than the official procedure and has slightly better sensitivity. E. C. A.

1F20. **Determination of Kjeldahl nitrogen in sugar beet and sugar-factory intermediate products.** Kubadinow, N.; and Roesner, G. *Sucr. Belge*, 1977, **96**, 9–16 (in French).—Comparison was made between Kjeldahl-N determination by (*i*) discontinuous distillation in a Technicon BD40 automatic digester system, and (*ii*) a colorimetric Auto-Analyzer method in which the N is determined as indophenol green. After reaction for 45 min, method (*i*) gave a conversion of betaine-N close to the theoretical value; with method (*ii*), only 60% of the betaine-N had been converted into NH_3, and the amount converted was still below the theoretical value after reaction for 2 h. An examination was made of possible causes of the low results, and attempts were made so to adjust conditions that the two methods gave similar results. The use of H_2O_2 or $HClO_4$ did not give an improvement in the results. Investigations were also made of the effects of specific catalysts on the reaction time needed for satisfactory conversion of betaine. Unsatisfactory results were obtained by using ZrO_2 as catalyst (Glowa, *J. Assoc. Off. Anal. Chem.*, 1974, **57**, 1228). All results indicated that reaction for >2 h was required, and the best results were given by a digestion mixture comprising H_2SO_4 (1 litre), K_2SO_4 (500 g) and HgO (25 g). With this mixture, the accuracy of method (*ii*) was improved so that results agreed to within 2·9% with those of method (*i*). Since H_2O_2 had no adverse effect, its use was recommended for reduction of floc formation in the test soln. INT. SUGAR J. ABSTR.

1F21. **Determination of ammonium ions in the sugar factory.** Devillers, P.; Detavernier, R.; and Groult, M. *Sucr. Fr.*, 1977, **118**, 66–73 (in French).—In a colorimetric method based on that of Fawcett and Scott (*J. Clin. Pathol.*, 1960, **13**, 156), 5 ml of an aq. soln. containing 2·6 ml l^{-1} of NaClO soln. and 5 g l^{-1} of NaOH is added to 0·2 ml of the test soln. (containing up to 200 mg l^{-1} of NH_4^+) in a 20-ml test-tube, followed by addition to 5 ml of an aq. soln. of 10 g l^{-1} of phenol and 0·051 g l^{-1} of Na_2-$[Fe(CN)_5NO].2H_2O$. The test-tube and contents are heated in a water bath for 30 min at 55°, and the absorbance of the soln. is measured at 550 nm. (An automated version of the method is also described.) Another method is based on use of an NH_4^+-specific electrode, which is inserted into a flask containing 50 ml of the test or standard NH_4Cl soln. to which

has been added 10 ml of a reagent prepared by dissolving 160 g of NaOH and 15 g of Na_2edta in H_2O and making the soln. up to 1 kg; the NH_4^+ value is read directly from a pH meter. The methods have been used to determine NH_4^+ and NH_3 in, *e.g.*, juices, press-water, cossettes and pulp. The automated method was also applied to amide determinations. INT. SUGAR J. ABSTR.

1F22. **Spectrophotometric determination of sugar content.** Gabor, E.; and Revesz, E. *Elelmiszeripari Foiskola, Szeged, Tud. Kozl.*, 1976, (6), 39–50 (in Hungarian).—The method is based on spectrophotometric measurement of the decrease in $CuSO_4$ concn. caused by pptn. of Cu_2O during reduction by sugar in the Bertrand reaction. A reaction mixture of similar composition, but not containing sugar, is used for the comparison. The method is much faster than the Bertrand determination and requires fewer chemicals, and statistical analysis of the results has shown it to be as accurate as the standard Bertrand sugar method.
HUNG. TECH. ABSTR.

1F23. **Determination of invert sugar in technical sugar juices. II. Comparative tests on various analytical methods with particular reference to thick juice control.** Reinefeld, E.; Emmerich, A.; van Malland, H.; and Miehe, D. (Inst. Landwirtschaftl. Tech. u. Zuckerind., Tech. Univ. Braunschweig, W. Germany). *Zucker*, 1976, **29** (12), 657–664 (in German).—The invert sugar contents of raw juice, thick juice and molasses were determined by (*i*) an iodimetric procedure (Spengler *et al.*, *Z. Wirtschaftsgruppe Zuckerind.*, 1936, **86**, 130, 322) and three colorimetric procedures (references and details given) involving the use of (*ii*) dinitrophthalic acid, (*iii*) triphenyltetrazolium chloride and (*iv*) 4-amino-3-hydrazino-5-mercapto-1,2,4-triazole as reagents, and the results were compared with a specific enzymic method. The results were all higher than those of the enzymic method, but were mutually comparable. Thick juices may be analysed satisfactorily only by methods (*ii*) and (*iv*), but formaldehyde interferes with method (*iv*), precluding its use on raw juices. Procedure (*iii*) lacks the sensitivity of the other two colorimetric methods. Modified titrimetric procedures in which the pptd. Cu^I was assayed iodimetrically, and in which the excess of Cu^{II} was determined complexometrically with edta, were compared with procedure (*i*) on samples of molasses; these contained reducing substances that had a marked effect on the consumption of iodine. No advantage was gained from these modifications. (For Part I see *Anal. Abstr.*, 1977, **33**, 1F6.)
D. W. S.

1F24. **Absolute determination of sucrose in mill and refinery products by isotope dilution. I. Isolation and purification of sucrose for counting.** Piper, N. R. (CSR Limited, O'Connell St., Sydney, N.S.W., Australia). *Int. Sugar J.*, 1977, **79** (942), 153–156.—The mixture of sample and [^{14}C]sucrose is initially clarified with basic Pb acetate, and the filtrate is 'de-leaded' by treatment with K oxalate - Na_2HPO_4 and further filtration. Sucrose is pptd. as 'Ba saccharate', recovered from the ppt. by carbonatation and further purified by treatment with activated carbon and ion-exchange resin (Amberlite MB-3). The purified and decolorised filtrate is concentrated to 50° Brix, poured into denatured abs. ethanol and set aside for 48 h for crystallisation of the sucrose (≃90% yield). The crystals are washed with ethanol and with acetone, recrystallised twice from denatured abs. ethanol and dried for 16 h in a vacuum oven at 50°. This purification procedure is free from bias and is unaffected by high concn. of impurities in the original sample (such as occur in, *e.g.*, molasses, mill mud and golden syrup).
E. C. A.

1F25. **Water determination in fondant [with a refractometer].** Gotsch, G. *Rev. Choc. Confect. Bakery*, 1977, **2** (1), 37.

1F26. **Measurement of total and gelatinised starch by glucoamylase [exo-1,4-α-glucosidase] and o-toluidine reagent.** Chiang, B.-Y.; and Johnson, J. A. (Dept. Grain Sci. and Ind., Kansas State Univ., Manhattan, Kans. 66502, U.S.A.). *Cereal Chem.*, 1977, **54** (3), 429–435.—In the rapid and precise method described for determining gelatinised starch in foods, an aq. dispersion of the sample is digested with glucoamylase for 30 min at 40°, glucoamylase is inactivated and pptd. (together with other proteins) by trichloroacetic acid, with centrifuging, and the glucose formed is determined spectrophotometrically at 630 nm after heating the supernatant soln. with *o*-toluidine soln. (in anhyd. acetic acid) for 10 min in a boiling-water bath, cooling, and adding anhyd. acetic acid. Total starch is determined similarly after gelatinisation of intact starch with NaOH soln. and neutralisation with HCl. Total starch, calculated by multiplying the result for glucose by 0·90, showed good agreement (correlation coeff. 0·98) with results by the official polarimetric method of the American Association of Cereal Chemists. E. C. A.

1F27. **Determination of the proportion of pectin and starch in their mixtures by infra-red spectroscopy.** Filippov, M. P.; Kaldare, G.; and Bondareva, O. V. *Izv. Akad. Nauk Mold. SSR, Ser. Biol. Khim. Nauk*, 1977, (1), 90–91 (in Russian).—The ratios of pectin to starch can be determined by measuring the ratios of their respective absorption values at 950 and 1150, 1080 and 1098, or 1098 and 1150 cm^{-1}, and referring the results to calibration graphs. For standard mixtures, measured at 1098 and 1150 cm^{-1}, the errors did not exceed ±3%. CHEM. ABSTR.

1F28. **Oligosaccharides released during hydration of textured soya as determined by high-performance liquid chromatography.** Havel, Eileen; Tweeten, Thomas N.; Seib, Paul A.; Wetzel, David L.; Liang, Yen Teh; and Smith, Oak B. (Dept. Grain Sci. and Ind., Kansas State Univ., Manhattan, Kans. 66506, U.S.A.). *J. Food Sci.*, 1977, **42** (3), 666–668.—Sucrose, raffinose and (in particular) stachyose were determined in prepared mixtures and in extracts of textured soya by h.p.l.c. on a stainless-steel column packed with Partisil 10 PAC protected from contamination by a pre-column of Co:Pell PAC. The mobile phase (70 ml h^{-1}) was acetonitrile - 2·2mM-$KHPO_4$ adjusted to pH 5 with dil. H_3PO_4, and detection was by refractometry. The method was used to determine the amount of stachyose released during hydration of the soya with boiling water. For stachyose, the coeff. of variation was ± 0·8% for a pure soln. (10 mg ml^{-1}) and ±1·4% for a soya extract. Recovery of stachyose added before boiling was 95%. R. F. S.

1F29. **Detection of foreign fats in dairy products. Part 2. Detection of vegetable fat by gas - liquid chromatography of sterols (reference method).** British Standards Institution (101 Pentonville Rd., London N1 9ND). BS 5475:Part 2:1977 [ISO

3594–1976]. Pp. 7.—The sterol digitonides are prepared as described in Part 1 (*Anal. Abstr.*, 1977, **33**, 4F16) and are dissolved in formamide - dimethylformamide (1 : 1). The liberated sterols are extracted into pentane and determined by g.l.c. at 220° to 250° on a U-shaped or coiled column (100 to 200 cm × 2 to 4 mm) packed with 2 to 4% of a methylsilicone gum rubber supported on an acid-washed and silanised flux-calcined diatomaceous earth (80 to 100 or 100 to 120 mesh), with N as carrier gas (30 to 60 ml min^{-1}) and a H-flame ionisation detector. The presence of a vegetable fat in the sample is indicated by a peak with the same retention time as β-sitosterol; this conclusion is supported by the appearance of peaks due to, *e.g.*, campesterol or stigmasterol. R. F. S.

1F30. **Automated measurement of D(−)- and L(+)-lactate in soya sauce and wine.** Kanbe, Chiyuki; Ozawa, Yoshinori; and Sakasai, Toshio (Cent. Res. Lab., Kikkoman Shoyu Co. Ltd., Noda-shi, Chibaken, Japan). *Agric. Biol. Chem.*, 1977, **41** (5), 863–867.—Total lactate was determined with a new carboxylic acid analyser S-603 (Nakajima *et al.*, *J. Chromatogr.*, 1976, **123**, 129). The standard error was <5%; one determination took 2 h. L(+)-Lactate was determined with a discrete-type autoanalyser (EEL Autochemist 171-S) with use of L(+)-lactate dehydrogenase; 30 samples could be determined in 1 h, and the standard error was <2%. Pre-treatment of the samples was unnecessary, apart from dilution, filtration or deproteinisation. E. A.

1F31. **Determination of pungency due to capsicum by gas - liquid chromatography.** Todd, P. H., jun.; Bensinger, M. G.; and Biftu, T. (Kalamazoo Spice Extraction Co., Kalamazoo, Mich. 49005, U.S.A.). *J. Food Sci.*, 1977, **42** (3), 660–665, 680.—The capsaicinoids were extracted from samples of fresh chillies, oleoresins, tinctures and plasters. Silyl derivatives were prepared by treatment with trifluoro-*NO*-bis(trimethylsilyl)acetamide in tetrahydrofuran medium for 5 min at room temp., then the mixture was submitted to g.l.c. on a column of 3% of SE-30 on Chromosorb G HP (100 to 120 mesh) temp.-programmed from 170° at 4° min^{-1} to 215° and maintained at 215° for 10 min; the carrier gas was N (20 ml min^{-1}), detection was by flame ionisation, and *N*-vanillyloctanamide was used as internal standard. The pungent capsaicinoid derivatives were eluted after the inactive constituents. A BASIC algorithm was used to calculate the percentages of individual capsaicinoids. Pungencies of the individual capsaicinoids were evaluated by using ASTA method 21.0 The actual (Scoville) pungency of each sample was calculated by multiplying the content of each capsaicinoid (capsaicin, nordihydrocapsaicin, dihydrocapsaicin, homocapsaicin and homodihydrocapsaicin) by the corresponding value of threshold pungency. R. F. S.

1F32. **Detection of colophony in asafoetida and compounded asafoetida by thin-layer chromatography.** Banerjee, T. S.; Mukherjee, D.; Guha, K. C.; Mathew, T. V.; and Roy, B. R. (Central Food Lab., Calcutta, India). *J. Inst. Chem. (India)*, 1976, **48** (6), 276–277.—The method involves detection of abietic acid (from the resin). The sample is triturated for 5 min with light petroleum (boiling-range 40° to 60°), and a portion of the filtered liquid is submitted to development on a layer of Silica gel G (activated at 100° for 1 h before use), with benzene - methanol (9 : 1) as mobile phase. After development for 10 cm, the chromatogram is dried in air, sprayed with CCl_4 - phenol (2 : 1) and exposed to Br vapour. Abietic acid appears as a blue spot (R_F 0·75) within 3 min, and is compared with a spot for authentic abietic acid (or an extract of the pure resin) on the same chromatogram. The detection limit is 5 μg. Materials extracted from wheat or rice flour, and fatty acids and sterols, do not interfere. B. H.

1F33. **Processes for extracting flavouring substances from flavoured food products.** Berner, Charles. *Ann. Falsif. Expert. Chim.*, 1976, **69** (746), 737–744 (in French).—With use of 30% sucrose soln. to simulate the food, experiments with various solvents and methods of extraction were carried out to extract several esters and other flavouring compounds. For extraction by shaking in a separating-funnel, CH_2Cl_2 was the best extractant, followed by $CHCl_3$, ethyl ether - pentane (2 : 1) and pentane. Recoveries were ≃70% at levels of 1 p.p.m. and 80 to 95% at 20 or 50 p.p.m. Steam-distillation at atmospheric pressure gave low recoveries, and artefacts were formed, but steam-distillation under reduced pressure (≃2 Torr) with a two-stage apparatus gave excellent results. Liquid - liquid extraction in various pieces of apparatus, or percolation, gave recoveries similar to those attained by using a separating-funnel; continuous percolation for >10 h caused decomposition. A table is presented listing types of foods and recommended solvent or method of extraction. CHEM. ABSTR.

1F34. **Examination of vanilla extracts and of products flavoured with these extracts.** Meili, M.; and Chaveron, H. (Dept. Genie Biol., Univ. Technol., Compiegne, France). *Ann. Falsif. Expert. Chim.*, 1976, **69** (746), 745–756 (in French).—For vanilla extracts, Microcel (synthetic calcium silicate) is added to the sample until a powder forms, and the powder is extracted in a Soxhlet apparatus with ethyl ether - pentane (7 : 3); for powdered vanilla extracts, acidified Microcel is used to prevent formation of acetals. For the flavoured products, a slurry of the sample is prepared in acidified aq. 10% ethanol, proteins are pptd. with $K_4Fe(CN)_6$ and $ZnSO_4$, and the aromatic components are extracted into H_2O. The aq. and organic extracts, after concentration, are either applied to t.l.c. plates or are trimethylsilylated, the derivatives being subjected to g.l.c. for determination of vanillin and 4-hydroxybenzaldehyde. In the t.l.c. procedures, aldehydes are separated on silica gel, with benzene - methanol (97 : 3) as developing solvent, the spots being located under u.v. radiation or by spraying with hydrazine reagent; acids and alcohols are separated on cellulose, with propanol - 8N-NH_3 (3 : 1) and detection by 4-nitroaniline - $NaNO_2$ reagent; 4-methoxybenzyl alcohol is isolated on silica gel, with $CHCl_3$ - ethyl acetate (9 : 10) and H_2SO_4 detection; and coumarin is isolated similarly to aldehydes, but is detected under u.v. radiation after spraying with alcoholic KOH. The g.l.c. is effected on a column packed with 10% of Carbowax 20M on Chemosorb, with N as carrier gas (30 ml min^{-1}) and flame ionisation detection. CHEM. ABSTR.

1F35. **Determination of vanillin by gas - liquid chromatography.** Repasi, M.; and Csont, M. *Elelmiszervizsgalati Kozl.*, 1976, **22** (3), 164–167 (in Hungarian).—After extraction of the sample with ether - ethanol (1 : 1), a 1 to 3-μl portion of the extract is subjected to g.l.c. at 170° on a column packed with 5% of XE-60 on Chromosorb W. Determination can be carried out, by measurement

of the peak heights, with an error of 5% with use of quinol as internal standard; the extraction efficiency is nearly 100%. HUNG TECH. ABSTR.

1F36. **Analysis of flavour constituents. I. Gas - liquid chromatographic determination of vanillin and coumarin in foods.** Kan, Teruo; Kamata, Kunihiro; Mori, Kenichiro; Nakamura, Yoshiaki; Ueta, Tadahiko; and Totani, Tetsuya. *Tokyo-toritsu Eisei Kenkyusho Kenkyu Nempo*, 1976, **27** (1), 106–111 (in Japanese).—The sample was treated with H_2SO_4 and ethanol, then extracted with CH_2Cl_2; the extract was applied to a column of Florisil, and elution was carried out with methyl formate. The eluate was analysed by g.l.c. on a conventional column, with flame ionisation detection. The average recoveries of vanillin and coumarin from chocolate, biscuits, ice-cream, cocoa powder and caramel ranged from 90 to 94·6%. The detection limits were 0·12 μg for vanillin and 0·05 μg for coumarin. CHEM. ABSTR.

1F37. **Analysis of flavour constituents. II. High-performance liquid-chromatographic determination of vanillin and coumarin in foods.** Kamata, Kunihiro; Kan, Teruo; Yamanobe, Hideo; Mizuishi, Kazuko; Yamazoe, Ritsuko; and Totani, Tetsuya. *Tokyo-toritsu Eisei Kenkyusho Kenkyu Nempo*, 1976, **27** (1), 112–117 (in Japanese).—The compounds were determined by h.p.l.c. at 20° on a column of Permaphase ETH, with hexane - ethanol (49 : 1) as mobile phase (0·75 ml min^{-1}) and detection at 254 nm. The calibration graphs for both vanillin and coumarin were rectilinear down to 0·05 μg, and the detection limits were 30 and 5 ng, respectively. The recoveries ranged from 92·6 to 100% for vanillin and from 90 to 98% for coumarin. Analysis of 52 samples by this method indicated the presence of vanillin in 25 samples and coumarin in 13. CHEM. ABSTR.

1F38. **Methods of determination of sodium glutamate. II. Canned soups, sauces, cooked dishes, seasonings and various aromas.** Armand, Paul; Abello, Georges; and Gayte-Sorbier, Andre (Fac. Pharm., Marseille, France). *Ann. Falsif. Expert. Chim.*, 1976, **69** (742), 545–556 (in French).—The t.l.c. and enzymic methods previously used for dried soups (Gayte-Sorbier *et al.*, *Ibid.*, 1975, **68**, 439) were successfully applied to other types of foodstuffs. An examination was also made of a variant of the enzymic method, with visible-region (instead of u.v.) spectrophotometry. This depends on the reduction of L-glutamate in the presence of glutamate dehydrogenase, reduction, by the NADH so formed, of iodonitrotetrazolium to yield a formazan, and measurement of the absorbance at 492 nm. The three methods give concordant results and can be applied to all kinds of foodstuffs. R. F. S.

1F39. **Methods of determination of sodium glutamate. III. Use of an automatic centrifugal analyser.** Arnoux, Andre; Abello, Georges; Gayte-Sorbier, Andre; and Armand, Paul (Inst. J. Paoli et I. Calmettes, Marseille, France). *Ann. Falsif. Expert. Chim.*, 1976, **69** (742), 557–562 (in French).—The enzymic method previously reported (Gayte-Sorbier *et al.*, *Ibid.*, 1975, **68**, 439) was adapted for use with the Centrifichem analyser (Roche S.A., Neuilly), with which 30 samples can be analysed consecutively. The results obtained agreed with those by the manual method (mean deviation −1·7%). (For Part II see preceding abstract.) R. F. S.

1F40. **Analytical characterisation of polyglycerol ester emulsifiers.** Schuetze, Ingeborg (Inst. Oel- u. Margarine-ind., Magdeburg, E. Germany). *Nahrung*, 1977, **21** (5), 405–415 (in German).—The emulsifiers are fractionated by column chromatography on silica gel, then individual fractions are analysed by g.l.c., which is also used for determining the alcohol and fatty acid components of the emulsifiers. Determination of the hydrophilic - lipophilic balance from the ratio of the saponification value of the emulsifier to the acid value of the fatty acids is proposed as a means of grading the emulsifiers. R. F. S.

1F41. **Rapid detection and estimation of extraneous ammonium sulphate in milk and effects of the added salt on certain physico-chemical properties of buffalo milk.** Mittal, S. B.; and Roy, N. K. (Natl. Dairy Res. Inst., Karnal, India). *Indian J. Dairy Sci.*, 1976, **29** (4), 283–289.—To 1 ml of milk contained in a test-tube are added 0·5 ml each of 2% NaOH, 2% NaClO soln. and aq. 5% phenol, and the mixture is heated for 20 s in a boiling-water bath. Formation of a blue soln., which rapidly deepens in colour, indicates extraneous $(NH_4)_2SO_4$. As little as 0·1% of $(NH_4)_2SO_4$ can be detected, even in the presence of other adulterants such as urea, glucose and sucrose. CHEM. ABSTR.

1F42. **Detection of pimaricin [natamycin] on the surface of cheese.** Frede, Wolfgang (Chem. u. Lebensmittel-Untersuchungsanst., Hamburg, W. Germany). *Milchwissenschaft*, 1977, **32** (2), 66–67 (in German).—The use of this antibiotic (**I**) to prevent the formation of moulds on the surface of cheese is no longer permitted in W. Germany. To detect **I**, samples were extracted with methanol, and the extracts were examined by spectrophotometry at 303 and 318 nm. The limit of detection was 100 ng ml^{-1} of **I**. The presence of **I** was confirmed by t.l.c. on Kieselgel F_{254} and by reversed-phase h.p.l.c. on LiChrosorb RP-8 (*cf.* Mechlinsky and Schaffner, *J. Chromatogr.*, 1974, **99**, 619). The limit of detection by h.p.l.c. was 20 ng per injection. R. F. S.

1F43. **Determination of residues of carbamate pesticides in foods. I. Literature survey of methods.** Kubacki, S. J. (Inst. Przem. Ferment., Warsaw, Poland). *Pr. Inst. Lab. Badaw. Przem. Spozyw.*, 1976, **26** (1), 101–108 (in Polish).—A review is presented, with 17 references. NUTR. ABSTR. REV.

1F44. **Determination of isohumulones in hop extract.** Balyakina, L. P.; Emel'yanova, Z. I.; and Leoninok, V. A. (Vses. Nauchno-Issled. Inst. Pivo Bezalkogol'noi Prom., Moscow, USSR). *Fermentn. Spirt. Prom-st.*, 1977, (3), 40–42 (in Russian).—The method involves dissolving the dry extract in water, measuring the absorbance at 275 nm, and computing the results by reference to a graph (rectilinear) prepared from standard soln. containing 0·8 to 8·0 mg dl^{-1} of isohumulones. CHEM. ABSTR.

1F45. **Rapid instrumental analysis of composition of wine.** Kaffka, K. J.; and Norris, K. H. (Central Food Res. Inst., Budapest, Hungary). *Acta Aliment. Acad. Sci. Hung.*, 1976, **5** (3), 267–279.—A preliminary study was made of two types of wine by spectrophotometry in the near-i.r. region. Absorbances were measured at 1·072, ≃1·450, 1·696, 1·816, ≃2·150 and 2·232 μm, and four physical parameters were derived from differences in absorbance at

pairs of wavelengths, from which chemical compositions were calculated by regression analysis for 26 prepared samples containing 11·88 to 17·47% (v/v) of ethanol, 8·34 to 88·47 g l^{-1} of sugar and 4·89 to 82·63 g l^{-1} of acid. The errors in predicting the composition from the i.r. analysis were $<0{\cdot}1\%$ for ethanol, <2 g l^{-1} for sugar and <1 g l^{-1} for acid. All the main components could be determined with the same sensor and instrument by use of different interference filters to isolate the required wavelengths. R. F. S.

1F46. **Automated analysis of beverages, wine in particular. II. Automatable colorimetric determination of total sulphurous acid in grape must and wine, compared with the official method.** Hieke, E.; and Kreisel, A. (Chem. Untersuchungsamt, Zollhafen 12, Mainz, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **285** (1), 39–42.—To 5 ml of wine are added 1 ml of NaCl - $HgCl_2$ soln. and 10 ml of 0·1N-NaOH, and the soln. is diluted to 100 ml. To a 5-ml portion of this soln. are added 5 ml of dil. formaldehyde soln. and 5 ml of a 0·04% pararosaniline soln. The soln. is diluted to 50 ml and the absorbance at 570 nm is measured after 8 min. The SO_2 content is read from a calibration graph. The influence of natural dyes is examined. The proposed method is compared with the official method [a modification of that of Reith and Willems (*Anal. Abstr.*, 1959, **6**, 2336)]. The coeff. of variation for the proposed and official methods are from $\pm 1{\cdot}27$ to $\pm 2{\cdot}4$ and from $\pm 7{\cdot}54$ to $\pm 1{\cdot}96$, respectively, for concn. between 47·5 and 380 mg l^{-1} of SO_2. (For Part I see *Anal. Abstr.*, 1974, **26**, 525.) J. K. C.

1F47. **Jaulmes' apparatus for steam-distillation of, *e.g.*, alcohol, volatile acids and ammonia.** Korszen, Ryszarda; and Krawczyk, Wladyslaw. *Przem. Ferment. Rolny*, 1977, **21** (2), 11–13 (in Polish).—The apparatus described incorporates a jacketted copper boiler, a water purifier and two glass assemblies each containing a bubbler, rectification column and condenser. Arrangements are made for automation of the process, and this permits the alcohol, volatile acids, NH_3 and other volatile substances to be distilled in 3 to 4 min. The apparatus was used to good effect in determining these compounds in fruit and non-fruit wines. CHEM. ABSTR.

1F48. **Colorimetric determination of volatile acids in wine.** Dima, Florica; and Ghimicescu, G. (Fac. Pharm., Iasi, Romania). *Ann. Falsif. Expert. Chim.*, 1977, **70** (749), 35–38 (in French).—The sample is treated with H_2O_2 and $NaAsO_2$, and the soln., after adjusting to pH 3 with tartaric acid, is steam-distilled in a micro-Kjeldahl apparatus. To the distillate is added NH_4VO_3 soln., and, after 20 min (to allow for formation of the yellow polyvanadyl complex), the absorbance is measured at 400 nm against a reagent blank; the result is calculated as g l^{-1} of H_2SO_4. The calibration graph (based on standard soln. of acetic acid) is rectilinear for up to 5 μmol of acetic acid in 0·2 ml of wine, and the results agree with those obtained by titration with $Ba(OH)_2$. R. F. S.

1F49. **Determination of volatile acidity in wines. Simplified technique with oxidation of sulphurous acid before steam-distillation.** Schneyder, Josef; and Pluhar, Gertrude (Landw.-Chem. Bundesversuchsanst., Wien, Austria). *Mitt. Hoeheren Bundeslehr Versuchsanst. Wein Obstbau, Klosterneuburg*, 1977, **27** (1), 14–17 (in German).—The samples were adjusted to pH $\simeq$10 with 2N-NaOH and treated with $FeCl_3$ - HCl soln. to oxidise SO_2, then steam-distilled. The acids in the distillate were titrated with 0·1N-NaOH (phenolphthalein as indicator) and calculated as acetic acid (either in m-equiv. l^{-1} or in g l^{-1}). R. F. S.

1F50. **Evaluation of the D(−)- and L(+)-lactic acid contents of wine.** Bandion, Franz; and Valenta, Maria (Landw.-Chem. Bundesversuchsanst., Wien, Austria). *Mitt. Hoeheren Bundeslehr Versuchsanst. Wein Obstbau, Klosterneuburg*, 1977, **27** (1), 4–10 (in German).—Samples were clarified by treatment with poly(vinylpyrrolidone) and diluted according to their expected lactic acid content. The D(−)- and L(+)-acids were then determined, with use of NAD and alanine aminotransferase, by a modification of the Boehringer Mannheim method. Recoveries of the added acids were satisfactory. R. F. S.

1F51. **Determination of sorbitol in wines by gas - liquid chromatography of its acetyl derivative.** Bertrand, A.; and Pissard, R. (Inst. Oenol., Univ. Bordeaux II, Talence, France). *Ann. Falsif. Expert. Chim.*, 1976, **69** (742), 571–579 (in French).—Acids and sugars in the sample are removed initially by adsorption on a column of Amberlite IRA 400 (OH^- form), H_2O being used to wash the column. Polyhydric compounds in the percolate are acetylated by treatment with acetic anhydride in pyridine soln., and are separated by g.l.c. at 210° on a stainless-steel column packed with 7% of QF-1 on Gas-Chrom Q, with flame ionisation detection. Sorbitol is determined from the peak height for its derivative relative to that for the derivative of the internal standard, perseitol. The calibration graph is rectilinear for up to 80 mg l^{-1} of sorbitol, and the reproducibility and recovery are satisfactory. R. F. S.

1F52. **Estimation of 2-furaldehyde, extract, permanganate time and pH in beverage spirits.** Fifth Report of the Research Committee on the Analysis of Potable Spirits. *J. Assoc. Public Anal.*, 1977, **15** (2), 47–54.—Inter-laboratory studies of methods for determining 2-furaldehyde and extract in whiskies, rums and brandies and of methods for permanganate time and pH of alcohol intended for the production of gin or vodka are reported. A g.l.c. procedure, preferably with use of an electronic integrator for measurement of detector response, is recommended for the determination of 2-furaldehyde; the AOAC u.v. procedure was found to give high results (owing to the presence of other u.v.-absorbing materials), and the official French method (with use of anilinium acetate) constitutes a toxic hazard. For extract, the AOAC procedure is recommended. The permanganate-time test is considered suitable only for manufacturers' and traders' internal quality-control purposes. For the determination of pH (primarily of interest for internal quality-control purposes), it is recommended that highly rectified spirit should be diluted to $\simeq$40% (v/v) with freshly boiled and cooled distilled water. E. C. A.

1F53. **Determination of the composition of fusel oils by gas chromatography.** Otvos, L.; and Szep, I. *Elelmiszervizsgalati Kozl.*, 1976, **22** (3), 142–150 (in Hungarian).—The g.l.c. method described, which was suitable for the routine determination of C_3 to C_5 alcohols (particularly 2- and 3-methylbutanol) in fusel oils, involved use of a stainless-steel column (3 m × 4 mm) packed with 25% of dodecyl phthalate on Celite and operated at 140°, with N (1·8 kg cm^{-2}) as carrier gas. Comparison of the results

with those obtained by a distillation method showed that only ≃60% of the 2- and 3-methylbutanol content was determined by the latter method.
HUNG. TECH. ABSTR.

1F54. **Rapid iodine value determination using mercury(II) acetate as accelerator.** Hashemy-Tonkabony, S. E. (Res. Lab., Dept. Food Hyg., Univ. Tehran, Iran). *J. Am. Oil Chem. Soc.*, 1977, **54** (6), 233.—In an attempt to reduce the reaction time from the standard 30 min, without loss of accuracy, a 2·5% soln. of Hg^{II} acetate in acetic acid is used as accelerator. The procedure is as usual, but the KI is added, and the subsequent titration is carried out, 1 or 3 min after addition of the Wijs or Hanus reagent, respectively. Results are satisfactory for butter, various vegetable oils and shortenings. E. A.

1F55. **Determination of pentane formed during autoxidation of oils contained in solid samples.** Bigalli, G. (Hershey Foods Corp., Res. Lab., Hershey, Pa. 17033, U.S.A.). *J. Am. Oil Chem. Soc.*, 1977, **54** (6), 229–232.—The sample, plus water and hexane (as carrier for the pentane), is distilled in an apparatus of the Dean and Stark type, and the dried (Na_2SO_4) hexane layer is subjected to g.l.c. in a stainless-steel column (8 ft. × 0·125 in.) packed with 15% of SE-30 on Chromosorb P AW (100 to 120 mesh), with temp.-programming from 80° (maintained for 4 min) to 230° at 20° min^{-1}. The carrier gas is N (40 ml min^{-1}) and detection is by flame ionisation. Standardisation is with a soln. of pentane in hexane. The sensitivity is 0·1 p.p.m. Samples included fats, nuts and chocolates. E. A.

1F56. **On what does the life of frying fats depend and how can the condition of the fat be determined analytically?** Arneth, W. (Inst. Chem. Phys., Bundesanst. Fleischforsch., Kulmbach, W. Germany). *Fleischwirtschaft*, 1977, **57** (4), 665–667 (in German).—A review is presented in which the changes occurring in fats during frying, and methods for evaluating the extent of fat degradation, are discussed. The smoke point, acid number, and the content of oxidised fatty acids insol. in light petroleum are considered to be parameters especially suitable for the evaluation. (10 references.)
CHEM. ABSTR.

1F57. **Determination of the smoke point for the evaluation of frying fats.** Arens, M.; Guhr, Gisela; and Waibel, J. (Bundesanst. Fettforsch., Muenster, W. Germany). *Fette, Seifen, Anstrichm.*, 1977, **79** (6), 256–259 (in German).—A method standardised for the determination of smoke point by a Working Group of the Deutsche Gesellschaft fuer Fettwissenschaft e.V. was evaluated in a collaborative study on three fat samples. Statistical evaluation of the results showed a discrepancy between the repeatability (9°) and the comparability (25°), but it should be possible to improve the comparability by, *e.g.*, intensive training with a standard sample, or by the development of automated end-point detection. R. F. S.

See also Abstracts: **1C62**, Testing of mineral oil that may come into contact with food. **1D10**, Determination of Ca, Ba and Sr in foods. **1D84**, Determination of triclosan in fish. **1D114**, Analysis of anchovy oil. **1D153**, Assay of lipoxygenase. **1E37**, Analysis of flavouring oils. **1E72**, Analysis of preservatives. **1G1**, Determination of N in protein. **1G4**, Determination of β-glucan in oat and barley gum. **1G18**, Determination of bromacil in fruit. **1G20**, Effects of cooking and processing on ethylenethiourea and the precursor fungicides. **1G24**, Determination of chloramphenicols in potatoes. **1H56**, Determination of COD of tomato waste.

G—AGRICULTURE

Including crops, soil, fertilisers, animal feeding-stuffs, and herbicides, fungicides and other pesticides

1G1. **Micro-determination of nitrogen in seed protein extracts with the salicylate - dichloroisocyanurate colour reaction.** Felker, Peter (Dept. Bot. and Plant Pathol., State Univ., East Lansing, Mich. 48824, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1080.—A method (based on Kjeldahl digestion and spectrophotometry at 660 nm) is described in which the colour development is carried out on the entire digest of the sample, thus avoiding dilutions, washings and transfers of soln. and hence enabling as little as 0·5 to 5 μg of protein-N to be determined.
G. P. C.

1G2. **Revised method for determining phosphate-phosphorus levels in sugar-beet-leaf petioles.** Varvel, G. E.; Peterson, G. A.; and Anderson, F. N. (Nebraska Agric. Exp. Stn., Lincoln, Nebr., U.S.A.). *J. Am. Soc. Sugar Beet Technol.*, 1976, **19** (2), 138–142.—The molybdophosphate method of Johnson and Ulrich [*Calif. Agric. Exp. Stn. Bull.*, 1950 (766), 25] with use of $SnCl_2$ as reductant (*i*) is compared with the same procedure but with use of ascorbic acid (*ii*) and also with a new method (*iii*) with ascorbic acid reduction. Method (*iii*) requires no H_2O_2 oxidation and thus needs only half the analysis time of (*i*). There was good agreement between results by (*i*) and (*ii*), but results by (*iii*) were consistently slightly low, although correlation with results by (*i*) was good. C. B.

1G3. **Rapid and simple method of screening rapeseed and mustard seed for erucic acid content.** McGregor, D. I. (Res. Stn., Agric. Canada, Saskatoon, Sask.). *Can. J. Plant Sci.*, 1977, **57** (1), 133–142.—Rapeseed oil or mustard seed oil was placed in a screw-capped tube with ethanol (3·5 ml) and heated to 70° to obtain a clear soln. The tube was then transferred to a water bath at 30° and the time taken for the soln. to turn completely opaque was noted. It was found that the higher the erucic acid content of the oil, the shorter the time required for the soln. to turn opaque. Calibration graphs were rectilinear. For max. sensitivity, it was found that 180 mg of oil per 3·5 ml of ethanol [or methanol - propanol (17 : 20)] was optimal. Results obtained for 22 *Brassica* cultivars were compared with those obtained by g.l.c. on butanediol succinate. The effects on solubility and fatty acid composition of three methods of extraction were also investigated.
A. B. K.-S.

1G4. **Determination of β-glucan in oats and barley.** Wood, P. J.; Paton, D.; and Siddiqui, I. R. (Food Res. Inst., Agric. Canada, Ottawa). *Cereal Chem.*,

1977, **54** (3), 524–533.—Glucose in oat or barley gum was determined by a modification of the Dische cysteine - H_2SO_4 reaction. The difference between this and the starch content, as measured enzymically, gave the β-glucan content of the gum. Procedures for the extraction, isolation and purification of the gum are described. For routine purposes, an adequate product was obtained by extraction with Na_2CO_3 - $NaHCO_3$ buffer soln. (pH 10) at 45° for 0·5 h and centrifugation (5 min at 24,000 *g*) (these steps being repeated twice), adjustment of the combined extracts to pH 4·5, further centrifugation (10 min at 24,000 *g*), heating at 100° for 10 min to inactivate enzymes, followed by dialysis (16 h against tap-water and 2 h against distilled water). This simplified procedure gave results that were significantly higher than those obtained by the method of Fleming *et al.* (*cf. Anal. Abstr.*, 1975, **28**, 4F44). Starch generally constituted <2% of the gum, so that omission of the determination of starch introduced only a small error. E. C. A.

1G5. **Note on a two-dimensional thin-layer chromatography procedure for determining aflatoxins in corn [maize].** Alexander, R. J.; and Baur, M. C. (Krause Milling Co., Milwaukee, Wis. 53201, U.S.A.). *Cereal Chem.*, 1977, **54** (3), 699–704.—Separation of aflatoxins from interfering substances in extracts of mouldy maize was achieved within ≃1 h on Brinkman SILG-HR-25 pre-coated plates (10 cm × 5 cm) by ascending-solvent development with acetone - $CHCl_3$ (3 : 22) and then (after drying the plate in air and turning it through 90°) denatured 95% ethanol. Visual comparison of zones with those for standards under long-wave u.v. radiation allows estimation of compounds to within ±20% of the actual amounts, as demonstrated by the results for, *e.g.*, 5, 10 or 15 p.p.b. of aflatoxins B_1, B_2, G_1 and G_2. E. C. A.

1G6. **Wavelength- and energy-dispersive X-ray micro-analysis with EMA [electron micro-probe analyser] and SEM - EDXRA [scanning electron microscope - energy-dispersive X-ray analyser] on thin sections of soils.** Bisdom, E. B. A.; Henstra, S.; Hornsveld, E. M.; Jongerius, A.; and Letsch, A. C. (Soil Surv. Inst., Wageningen, Netherlands). *Neth. J. Agric. Sci.*, 1976, **24** (4), 209–222.—The performance of these instruments was compared for analyses of organic matter, minerals and Fe - Mn nodules in soil sections. The detection of light elements (*e.g.*, N, O and C) in organic matter with the EMA gave poor results, and the SEM - EDXRA combination only detected heavier elements. The EMA produced somewhat better X-ray images of heavier elements, especially from an Fe - Mn nodule. However, with organic materials, the SEM - EDXRA X-ray images were similar to or slightly better than those by EMA. The max. magnification by SEM - EDXRA is ×10,000; that by EMA is ≃ ×500.
CHEM. ABSTR.

1G7. **Cadmium-ion activity in soils: evaluation of cadmium-ion-selective electrode.** Jackson, Danny R.; and Bondietti, Ernest A. (Environ. Sci. Div., Oak Ridge Natl. Lab., Tenn. 37830, U.S.A.). *Environ. Sci. Technol.*, 1977, **11** (6), 585–587.—An Orion 94–48 electrode is used to measure Cd^{2+} extracted from soil by 0·05M-$Ca(NO_3)_2$. Interference is minimised by rinsing the electrode with 0·01M-edta or by cleaning the surface mechanically, but the electrode cannot be used in the presence of high concn. of Pb^{2+}, Hg^{2+}, Cu^{2+} or Ag^+. Comparative analyses by a.a.s. and the electrode method showed good agreement for 5 to 100 p.p.m. of Cd. P. C. R.

1G8. **Modified curcumin method for boron analysis of soil extracts.** Kowalenko, C. G.; and Lavkulich, L. M. (Soil Res. Inst., Central Exp. Farm., Ottawa, Ont., Canada). *Can. J. Soil Sci.*, 1976, **56** (4), 537–539.—The method of Uppstrom (*Anal. Abstr.*, 1970, **18**, 2279) is shown to be suitable for the routine determination of up to 10 p.p.m. of B in soil. Recovery of 0·9 p.p.m. of B added to soil extracts is satisfactory. Fluoride interferes if present in amounts >20 times that of B, and NO_3^- interfere when the ratio of NO_3^- to B is >125 : 1; however, interference from 500 p.p.m. of N can be prevented by drying the sample at 65° to 70°. There is no interference from Fe^{II} (1000 p.p.m.) or Fe^{III} (100 p.p.m.). B. H.

1G9. **Determination of boron in soils and plants by the azomethine H [4-hydroxy-5-salicylideneamino-naphthalene-2,7-disulphonic acid] method.** Sippola, Jouko; and Ervio, Raimo (Inst. Soil Sci., Agric. Res. Centre, Vantaa, Finland). *Finn. Chem. Lett.*, 1977, (4–5), 138–140.—Boron is extracted from a 25-ml sample of finely sieved soil by boiling with a mixture of 50 ml of H_2O and activated charcoal and filtering, or from plant tissue (*e.g.*, barley or clover) by ashing a 1-g sample at 450° and extracting the ash with 10 ml of 0·1M-HCl. In each instance, a 2-ml portion of the extract is mixed with 4 ml of buffer masking soln. (250 g of ammonium acetate, 15 g of Na_2edta, 400 ml of H_2O and 125 ml of anhyd. acetic acid) and 4 ml of the reagent (0·9 g of azomethine H and 2 g of ascorbic acid in 200 ml of H_2O), and, after 1 h, the absorbance of the soln. is measured at 420 nm. Recoveries of B (1 to 2·5 mg l^{-1}) added to plant extracts ranged from 94 to 108%; the results were similar to those obtained with use of carmine reagent. P. C. R.

1G10. **Determination of nitrate in soil pastes by ion-selective electrodes.** Bound, Geoffrey B. (Macaulay Inst. Soil Res., Craigiebuckler, Aberdeen, Scotland). *J. Sci. Food Agric.*, 1977, **28** (6), 501–505.—An Orion nitrate exchanger (92–07) imbedded in poly-(vinyl chloride) was calibrated in unbuffered KNO_3 soln., and selectivity ratios between NO_3^- and the ions most likely to interfere were determined. The electrode was then pushed into pastes made from field-moist or air-dried soil and satd. $CaSO_4$ soln., and the potentials were measured. A s.c.e. with a 0·1M-KF bridge and a ceramic-frit liquid junction and a silver - AgCl wire in mM-KCl contained by a non-porous liquid junction were assessed as reference electrodes by comparing the NO_3^- values (E; mg l^{-1} of nitrate-N in the soil soln. at saturation) with those determined by conventional KCl extraction and colorimetric analysis (P; mg kg^{-1} of nitrate-N in the dry soil). The first reference electrode was adequate for dried samples, but not for moist ones, whereas the second was satisfactory for both types of sample, giving the equation $E = 2{\cdot}84 + 0{\cdot}45$ ($\pm 0{\cdot}03$)P. The regression coeff. was 0·863 (n = 60). T. H. N.

1G11. **Stabilised molybdenum-blue method for colorimetric determination of micro and semi-micro amounts of phosphorus [*e.g.*, in soils and plant material].** Nikolov, N. N. (N. Pushkarov Inst. Soil Sci. and Programming Crops, Sofia, Bulgaria). *Pochvozn. Agrokhim.*, 1976, **11** (6), 32–40 (in Bulgarian).—The sample soln., containing 1 to 60 μg of P,

is adjusted to pH 3 with 2M-NaOH and M-H_2SO_4 in the presence of 2,4-dinitrophenol, and then treated with 3 ml of a soln. of $(NH_4)_6Mo_7O_{24}.4H_2O$ (16·67 g) in 3·35M-H_2SO_4 (to 1 l). The sample soln. is diluted with H_2O to 45 ml and treated with 2 ml of a reducing soln. prepared by dissolving 0·075 g of $SnCl_2.2H_2O$ and 0·5 ml of HCl in 50 ml of H_2O, adding 0·75 g of ascorbic acid, and diluting the mixed soln. to 100 ml. This soln. is usable after 15 min and can be kept for 1 day. The absorbance of the soln. is measured at 715 nm after 6 min. The colour is stable for 8 h, and Beer's law holds for 0·02 to 1·3 μg ml^{-1} of P in the final soln.

G. S. S.

1G12. **Gas-chromatographic (electron-capture detector) determination of anthracene in soil and sediment.** Tatsukawa, Ryo; Kawano, Masahide; and Wakimoto, Tadaaki (Dept. Environ. Prot., Coll. Agric., Univ., Matsuyama-shi, Ehime, Japan). *Bunseki Kagaku*, 1977, **26** (6), 412–416 (in Japanese).—The sample of soil is mixed with ethanolic KOH and digested under reflux for 3 h. After cooling to room temp. the mixture is extracted with hexane. The extract is transferred to a column of Florisil, which is washed with hexane, and anthracene (plus phenanthrene) is eluted with hexane - ethyl ether (19 : 1). The eluate is concentrated and a portion is analysed for anthracene and phenanthrene by g.l.c. A second portion is shaken with H_2SO_4 to remove the anthracene then the phenanthrene is determined by g.l.c. In each instance, g.l.c. is carried out at 150° on a column (2 m × 3 mm) packed with 2% of DEGS on Chromosorb W-AW DMCS (80 to 100 mesh), with N (80 ml min^{-1}) as carrier gas. The detection limit for anthracene (determined by difference) was 5 ng g^{-1} and the recovery was >91%. The method has been applied to mineral soils and marine sediments with anthracene contents up to 81 ng g^{-1}.

1G13. **Determination of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin in environmental samples by high-resolution gas chromatography and low-resolution mass spectrometry.** Buser, Hans Rudolf (Swiss Fed. Res. Station, Waedenswil, Switzerland). *Anal. Chem.*, 1977, **49** (7), 918–922.—The sample-preparation procedure for soils and plants involved extraction with hexane - acetone (1 : 1), aq. partition of the extract under basic and neutral conditions, followed by clean-up on Celite - H_2SO_4 - silica gel, chromatography on alumina, and dissolution in tetradecane. The final extracts were analysed on columns (20 to 30 m × 0·35 mm) coated with OV-101 or OV-17 and operated at 207° to 220°, with He as carrier gas (35 cm s^{-1}) and electron-impact m.s. detection, the ions at *m/e* 320, 322 and 324 being monitored. Recoveries of the dioxin (down to 0·5 ng) added to environmental samples were >80%. G. P. C.

1G14. **Pyro[lysis - gas] chromatography as means of identification and differentiation of complex substances.** Aranda, V. G.; Osacar, J.; and Revuelta, G. (Inst. Nac. Carbon, Dept. de Zaragoza, Spain). *Quim. Anal.*, 1977, **31** (1), 9–17 (in Spanish).—The procedure involves pyrolysis of the sample (preferably with a Curie-point pyrolyser) and separation of the pyrolysis products by g.l.c., the resulting chromatogram being used as a 'finger-print' for identification. The effects of time and temp. of pyrolysis on the nature of the chromatogram are discussed, and results obtained for humic acids are reported. L. A. O'N.

1G15. **Heavy metals in agricultural lands receiving chemical sewage sludges. IV.** Van Loon, J. C. *Environ. Canada, Res. Progr. Abatement Munic. Pollut., Res. Rep.*, No. 51, 1976. Pp. 42.—A review is presented of existing and newly developed methods for sewage sludge analysis that are designed to give information on the form of metals in waste samples. Sludges can be dry-ashed at 450° without loss of Cd, Pb or appreciable amounts of Zn, but dry-ashing should not be used when Al is to be determined. There is indication that incineration at temp. usually used will release large amounts of As, Cd, Pb, Hg and Zn. Particular attention is paid to flameless atomic-absorption methods for V and Ag, development of methods for metal speciation studies in sludges, g.c. investigations, and liquid chromatography - atomic absorption studies.

WRC Inf.

1G16. **Trace analysis of oestradiol in animal chow by electron-capture gas chromatography.** Bowman, Malcolm C.; and Nony, Charles R. (Dept. Health, Educ. and Welfare, Food and Drug Adm., Natl. Center for Toxicol. Res., Jefferson, Ark. 72079, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (5), 160–163.—Oestradiol is extracted from the sample by a modification of the method used for stilboestrol by King *et al.* (*Anal. Abstr.*, 1977, **33**, 3G16), and is determined in the final eluate by g.l.c. (as its pentafluoropropionyl derivative) at 235° on 5% of OV-25. The limit of detection is 3 p.p.b. and recoveries at the 10- and 100-p.p.b. levels were 70 and 86·5%, respectively. M. J. Sl.

1G17. **Simultaneous determination of ethinyloestradiol and trenbolone acetate in compound feeds by combination of thin-layer chromatography and gas - liquid chromatography.** Wal, J. M.; Peleran, J. C.; and Bories, G. (Lab. Rech. Additifs Aliment., I.N.R.A., Toulouse, France). *J. Chromatogr.*, 1977, **136** (1), 165–169 (in French).—The steroids are extracted from the finely ground feeds by homogenisation with $CHCl_3$, and the lipids are eliminated by partition between hexane and acetonitrile. Each acetonitrile phase is evaporated *in vacuo* and the residue is dissolved in $CHCl_3$. The $CHCl_3$ soln. is applied as a band to a Kieselgel G plate (0·25 mm), and the chromatogram is developed with hexane - ethyl ether - CH_2Cl_2 (4 : 3 : 2). The combined steroid band is located in 254-nm radiation, and the silica, including that from 0·5 cm on each side of the band, is removed from the plate and extracted with $CHCl_3$. The $CHCl_3$ extract is analysed by g.l.c. on a Pyrex glass column (1·5 m × 0·25 in.) packed with 3% of SE-30 on Supelcoport (80 to 100 mesh) and operated isothermally at 240° for 3 min, temp.-programmed at 5° min^{-1} to 290°, and maintained at 290° for 5 min, with N as carrier gas (30 ml min^{-1}) and flame ionisation detection. The concn. of ethinyloestradiol (**I**) and trenbolone acetate (**II**) are determined by comparison with a standard mixture of **I** and **II** of similar concn. By the use of tritiated steroids the precision and accuracy of the t.l.c. process were shown to be high, and hence the use of labelled compounds is unnecessary for the determination. The standard deviation and coeff. of variation of the method at the 2-p.p.m. level were 0·057 and 2·8%, respectively, for **I**, and 0·076 and 3·8%, respectively, for **II**. R. F. S.

1G18. **Comparative study on analysis of the herbicide bromacil using electron-capture detection gas chromatography and mass fragmentography.** Suzuki, Tateo; Umedzu, Kimio; Itagaki, Yasuhiro; and

Tuzimura, Katura (Lab. Anal. Chem., Fac. Agric., Tohoku Univ., Sendai, Japan). *Agric. Biol. Chem.*, 1977, **41** (5), 775–779.—The extraction of bromacil from soil, mandarin oranges and murine tissue with ethyl acetate - H_2O (1 : 1), followed by clean-up on a Florisil column, was compared with earlier methods (Leistra *et al.*, *Weed Res.*, 1975, **15**, 243; Jolliffe *et al.*, *J. Agric. Food Chem.*, 1967, **15**, 174). Bromacil was determined with use of a glass column (1·2 m × 4 mm) packed with 5% of XE-60 on Gas-Chrom Z (60 to 80 mesh) and operated at 220° with N as carrier gas and a ^3H electron-capture detector. The results showed that the clean-up procedure was necessary in the analysis of soils. Mass fragmentography was also used for the determination, and seemed to be promising; the detection limit was 1 ng ml^{-1}. E. A.

1G19. **High-pressure liquid chromatographic analysis of oryzalin and other selected herbicides.** Kennedy, J. H. (Tippecanoe Lab., Eli Lilly and Co., Lafayette, Ind. 47902, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (2), 79–81.—A soln. of the herbicide in acetonitrile - H_2O is analysed on a column (300 mm × 4 mm) of μBondapak C_{18}, with 55% aq. acetonitrile as eluent (2 ml min^{-1}). Tebuthiuron is determined on a column of the same dimensions of μBondapak CN with 1% acetonitrile in 1-chlorobutane as eluent (1 ml min^{-1}). In both cases the eluate is monitored at 254 nm. The results compare well with those of a column - spectrophotometric assay. M. J. Sa.

1G20. **Ethylenethiourea.** International Union of Pure and Applied Chemistry, Applied Chemistry Division, Commission on Terminal Pesticide Residues. *Pure Appl. Chem.*, 1977, **49** (5), 675–689.—This report on the occurrence of ethylenethiourea (imidazolidine-2-thione; **I**) resulting from the agricultural use of ethylenebis(dithiocarbamate) fungicides has sections on analytical methodology (*e.g.*, t.l.c., polarography and g.l.c.); chemistry of **I**, including means of formation and metabolism in soil, plants and animals; dynamics and transformation of **I** residues; and effects of cooking and processing on **I** and the precursor fungicides. It is concluded, *inter alia*, that ethylenebis(dithiocarbamate) fungicides are acceptable for use in accordance with good agricultural practice; recommendations are made with regard to further investigations. R. L. J.

1G21. **Determination of benomyl residues in soil and in leaves and fruit of tomato plants.** Bazhanova, N. V.; Papoyan, F. A.; and Avetisyan, K. V. (Inst. Zashch. Rast., Erevan, Arm. SSR). *Biol. Zh. Arm.*, 1977, **30** (1), 86–90 (in Russian).—Benomyl is extracted from plant and soil samples with $CHCl_3$, and the extract is purified by treatment with active carbon, dried over Na_2SO_4 and evaporated to 0·1 ml. Pigments are decolorised by treating the concentrate with H_2SO_4 and, after 2 to 4 h, making the mixture strongly alkaline with 50% NaOH soln., and the cooled mixture is extracted with $CHCl_3$. This extract is evaporated to 0·1 ml and submitted to t.l.c. on Silufol sheets, with benzene - acetic acid - ethanol (14 : 7 : 3) as mobile phase and ethanolic 1% molybdophosphoric acid as spray reagent. Another method in which the benomyl is 'fixed' on the sheet by halogenation is also described. The sensitivity is 1 μg. Chem. Abstr.

1G22. **Chromato - polarographic determination of citrulin [diethyl 4-methyl-1,3-dithiolan-2-ylidenephosphoramidate] and tsiulin [phosfolan] in plants.** Bublik, L. I.; Safa, Muhommed Rifaat; and Kosmatyi, E. S. *Ukr. Khim. Zh.*, 1977, **43** (4), 418–419 (in Russian).—The sample (100 g) is shaken for 1 h with 150 ml of $CHCl_3$, the mixture is filtered, the residue is washed with $CHCl_3$ (4 × 10 ml), and the filtrate and washings are evaporated to 0·5 to 0·7 ml. This concentrate and a standard soln. are applied to the same plate [consisting of a layer of silica gel KSK - alumina (1 : 1) with $CaSO_4$ as binder], and development is carried out with hexane - acetone (1 : 1). The standard is located by successive application of 0·25% fluorescein soln. in dimethylformamide - ethanol (1 : 50) and a soln. prepared by dissolving 1·7 g of $AgNO_3$ in 10 ml each of H_2O and aq. NH_3 and 0·6 ml of aq. H_2O_2 and diluting to 200 ml with H_2O, and then examination in u.v. radiation. The corresponding zone from the untreated part of the chromatogram is placed in a polarographic cell containing phosphate buffer soln. (pH 6·9) in 10% ethanol as supporting electrolyte, and a polarogram is recorded; the $E_{\frac{1}{2}}$ value for either pesticide is −1·2 V *vs.* the s.c.e., and the sensitivity in either instance is 1 μg in 3 ml of electrolyte. G. S. S.

1G23. **Potentiometric determination of anionic surface-active substances in pesticides.** Ciocan, Nicolae; and Baiulescu, George E. *Rev. Chim. (Bucharest)*, 1977, **28** (5), 477–482 (in Romanian).—To prepare the indicator electrode, bubble air for 10 h through an ethanolic soln. of Co^{II} acetate, diphenylglyoxime and 1,10-phenanthroline (molar ratio 1 : 2 : 2), filter, dilute to be 10mM in Co, add an equal vol. of mM-Na decyl sulphate, extract the resulting Co complex (**I**) into 1,2-dichlorobenzene, dry the extract with Na_2SO_4 and dilute with the solvent to be mM in Co. Place the soln. in the reservoir of an electrode with a graphite membrane, and wash the electrode successively with 4mM-hexadecyltrimethylammonium bromide (**II**), 4mM-Na decyl sulphate and H_2O, and dry it with filter-paper. The electrode is used to titrate anionic surfactants potentiometrically with 4mM-**II** containing 4% of isopropyl alcohol at pH 1 to 11; the titrand may also contain isopropyl alcohol to increase the solubility of the surfactant. Inorganic anions and other ingredients of detergent compositions (*e.g.*, edta, borates and phosphates) do not interfere. The procedure can be used for the differentiating titration of mixtures of homologues, *e.g.*, of alkyl sulphates, or of different types of anions, *e.g.*, dodecylbenzene-mono- and -di-sulphonates. C. D. K.

1G24. **Determination of chloramphenicol residues [in potatoes] by gas chromatography.** Seefeld, F.; and Dunsing, Marianne (Inst. Pflanzenschutzforsch., Akad. Landwirtschaftwiss., Kleinmachnow, E. Germany). *Monatsh. Veterinaermed.*, 1976, **31** (18), 703–707 (in German).—The method was devised for determining chloramphenicol (**I**) applied during treatment to prevent rotting of stored potatoes. Details are given for the extraction of **I** from the sample into acetonitrile and for its conversion into a bis(trimethylsilyl) derivative, which is submitted to g.l.c. at 200° on a glass column packed with 3% of DC-200 on Varaport 30 (80 to 100 mesh) or 3% of SE-54 on Chromosorb G AW DMCS (80 to 100 mesh), with ^3H electron-capture detection. The limit of detection was 5 p.p.b. in the potatoes. The method was also applied to water, for which ethyl ether was used as extractant. G. E. F.

See also Abstracts: **1C46**, Analysis of a herbicide intermediate. **1C105**, Determination of polychlorinated biphenyls. **1D5**, Determination of

elements in plant material. **1D11**, Determination of Ca in plant material. **1D12**, Determination of ^{45}Ca in soil. **1D93**, **1D94**, Determination of pesticides in biological materials. **1E45**, Determination of lasalocid in, *e.g.*, feeds. **1F17**, **1F18**, Determination of aflatoxins. **1F20**, Determination of N in sugar beet. **1F43**, Determination of pesticide residues: review. **1H20**, Determination of 1,3,5-triethylhexahydro-1,3,5-triazine. **1H40**, Determination of SO_4^{2-} in soil. **1H52**, **1H53**, Determination of pesticides in water. **1J3**, Grinding of plant material. **1J7**, Extraction of pesticides. **1J65**, Determination of B in fertilisers. **1J67**, Determinations of Pb, Sn and Zn in soil.

H—AIR; WATER; EFFLUENTS

1H1. **Development and testing of portable wind-sensitive directional air sampler.** Deyo, J.; Toma, J.; and King, R. B. (Lewis Res. Center, Natl. Aeronautics and Space Admin., Cleveland, Ohio 44135, U.S.A.). *J. Air Pollut. Control Assoc.*, 1977, **27** (2), 142–144.—A design is presented for an air sampler that separates particulate matter into eight groups according to eight wind directions. After operating for up to 30 h, the apparatus provides samples, on Whatman No. 41 paper, for element or compound analysis. P. C. R.

1H2. **Comparison of cotton-dust sampling techniques.** Bethea, Robert M.; and Morey, Philip R. (Inst. Environ. Technol., Texas Tech. Univ., Lubbock, Tex. 79409, U.S.A.). *Am. Ind. Hyg. Assoc. J.*, 1976, **37** (11), 647–654.—A high-volume sampler (Environmental Protection Agency), a 6-stage sequential cascade impactor (Andersen 2000 Inc.) and modified vertical elutriators were compared. The total dust concn. as measured by the Andersen sampler was $\simeq$40% lower than that measured by either of the other two types of sampler, and it was concluded that sample loss and fibre bridging over the stage orifices in the Andersen sampler make this instrument of limited value in such sampling. M. W. G. B.

1H3. **Application of optical microscopy to studies of dust filtration. II. Specimen preparation techniques for analysing the distribution of dusts in filter fabrics.** Rothwell, E.; and Bickham, D. R. (Dept. Chem. Eng. and Fuel Technol., Univ. Sheffield, England). *J. Microsc. (Oxford)*, 1976, **108** (2), 211–219.—The difficulties of preparing representative sections, for microscopic examination, of dust cakes supported on filter fabrics by impregnation with resins are discussed. An evaporation - condenser assembly is described in which the specimen is placed in a thermal gradient and a volatile monomer (methyl 2-cyanoacrylate) is condensed from the vapour phase on to the dust particles and fabric fibres and is then polymerised *in situ* by exposure to NH_3 vapour. The particles and fibres are thus fixed in place, and representative samples can be obtained by vacuum impregnation with cold-setting resins and then sectioning by established techniques. M. W. G. B.

1H4. **Instrumental methods in the field of air-pollution analysis.** Louw, C. W. (Nat. Phys. Res. Lab., C.S.I.R., Pretoria, S. Africa). *Chemsa*, 1977, **3** (1), 2–3, 13.—A review is presented, with 30 references. M. J. St.

1H5. **Measuring, testing and analysing instruments for gases and vapours causing atmospheric pollution.** Anon. *Wasser, Luft Betr.*, 1976, **20** (10), 530–534 (in German).—The application and operation of commercial continuous and automatic indicating, monitoring, signalling and controlling equipment available in Germany are reviewed and discussed. S. E. P.

1H6. **Continuous mass-spectrometric analysis of particulates by use of surface ionisation.** Davis, William D. (General Electric Co., Res. and Dev. Center, Schenectady, N.Y. 12301, U.S.A.). *Environ. Sci. Technol.*, 1977, **11** (6), 587–592.—Real-time analysis of air-borne particulate matter is achieved by causing the particles to impinge on a rhenium ribbon electrically heated at $\simeq$1700°. The ions produced by surface ionisation are analysed by m.s. Particles containing 1000 atoms of a given element can be detected, but the technique is limited to elements (*e.g.*, Li, Sr, U, Cr, Pb and Cu) with ionisation potentials $<$8 eV; thus, Hg, Cd, Zn, As and Be cannot be measured. P. C. R.

1H7. **Continuous mass-spectrometric determination of concentration of particulate impurities in air by use of surface ionisation.** Davis, William D. (General Electric Co., Res. and Dev. Center, Schenectady, N.Y. 12301, U.S.A.). *Environ. Sci. Technol.*, 1977, **11** (6), 593–596.—The assembly described in the preceding abstract is used, with the rhenium ribbon at 830° to 920°. The best sensitivity is attained by collecting the particles on a 'cool' ribbon (with the heating current switched off) for 1 min, then 'flash-heating' the ribbon; this permits detection of as little as 0·1 pg m^{-3} of, *e.g.*, Pb or U. Samples have been analysed for Li, Na, K, Rb, Cs, Sr, U, Pb, Cr and Cu. Organic compounds can also be detected, but not individually identified. P. C. R.

1H8. **Method for the determination of tritium activity in air from nuclear-power plants.** Durcak, Herbert (Kernkraftwerk RWE-Bayernwerk G.m.b.H., Gundremmingen, Donau, W. Germany). *At. Strom.*, 1977, **22** (6), 144–145 (in German).—The air was passed (at 10 m^3 h^{-1}) through a plastic vessel in which water (containing the ^3H) was absorbed by 40 ml of 96% H_2SO_4 in a beaker. After the desired period (*e.g.*, when the vol. of liquid in the beaker had increased to 70 ml), the liquid (containing the ^3H as $^3H_2SO_4$) was diluted with an equal vol. of water and neutralised to phenolphthalein with anhyd. K_2CO_3. The resulting liquid was filtered and distilled, and ^3H in the distillate was measured by liquid scintillation counting. G. R.

1H9. **Determination of hydrogen cyanide in fire gases.** Tsuchiya, Yoshio; and Sumi, Kikuo (Fire Res. Sect., Div. Building Res., Natl. Res. Council, Ottawa, Ont., Canada). *J. Combustion Toxicol.*, 1976, **3** (4), 363–370.—Several methods of determination are briefly discussed and their susceptibility to the concn.-decrease effect (loss of HCN because of protracted sampling procedures, filtering, transferring and storing and because of adsorption on surfaces of the apparatus) and to interferences is discussed. Colorimetric methods and methods with use of ion-selective electrodes require less manipulation than g.l.c. methods and loss of HCN is thus minimised; however, they are susceptible to interference from other gases. Determination of HCN by

g.l.c. is likely to result in the greatest loss of HCN; nevertheless, it is possible to measure low levels even in the presence of other fire gases (*e.g.*, H_2O, CO, CO_2, and C_2N_2) by the use of a glass column packed with Porapak Q and a flame ionisation detector operated in the N mode. The ranges of HCN concn. as determined by 12 methods are listed. It is concluded that at least two different methods should be used in order that errors caused by adsorption and interference be eliminated. (15 references.)
J. P. A.

1H10. **Determination of sulphur dioxide in air.** Budesinsky, B. W. (Phelps Dodge Corp., Morenci, Ariz. 85540, U.S.A.). *Microchem. J.*, 1977, **22** (1), 55–59.—Air samples (50 l containing 0·3 to 19 mg of SO_2) were passed at 1 to 2 l min^{-1} through a 0·8-μm cellulose filter and scrubbed with aq. 80% isopropyl alcohol (15 ml) at 0° to remove H_2SO_4. The SO_2 was then absorbed in 0·3N-H_2O_2 at 0° using two midget impingers (15 ml in each). The absorbant was diluted to 50 ml and to a 10-ml aliquot were added acetone (20 ml) and 0·1% chlorophosphonazo III [3,6-bis-(4-chloro-2-phosphonophenylazo)-chromotropic acid] soln. (5 drops), then the pH was adjusted to 3 to 4. The soln. was titrated with 5mM-$Ba(ClO_4)_2$ (colour change from reddish or violet to blue). The method is compared with a gravimetric method and one with use of thoron I. T. G. A.

1H11. **Investigation of new sorbents for chromatographic - mass spectrometric determination of traces of volatile organic substances in the atmosphere.** Isidorov, V. A.; Zenkevich, I. G.; and Ioffe, B. V. (A. A. Zhdanov Leningrad State Univ., USSR). *Dokl. Akad. Nauk SSSR*, 1977, **235** (3), 618–621 (in Russian).—The g.c. properties of Carbopak B, Tenax GC, Polysorbimide 1 (a polymeric condensation product of pyromellitic anhydride and oxydianiline), Carbochrom K-5 and Silica gel C-4 have been examined with use of a katharometer detector and He (30 ml min^{-1}) as carrier gas. The carbon-black-based sorbents (Carbochrom K-5 and Carbopak B) adsorb low-mol.-wt. polar compounds only weakly, but show high affinity for alkanes and aromatic hydrocarbons. The porous polymers (Tenax GC and Polysorbimide 1) adsorb polar compounds more strongly than do the carbonaceous sorbents; *e.g.*, Polysorbimide 1 has been used effectively in the g.c. of 55 compounds in a 10-l sample of urban air. M. R. M.

1H12. **Chemiluminescence measurement of reactivity-weighted ethylene-equivalent hydrocarbons.** Black, Frank M.; High, Larry E.; and Fontijn, Arthur (Environ. Prot. Agency, Environ. Sci. Res. Lab., Research Triangle Park, N.C. 27711, U.S.A.). *Environ. Sci. Technol.*, 1977, **11** (6), 597–601.—In connection with analysing air for motor-vehicle emissions, a reactive-hydrocarbon analyser based on the chemiluminescence reaction of hydrocarbons with atomic O is described. This device provides rapid indication of the ethylene-equivalent concn. (*i.e.*, the concn. of ethylene that would give the same mass-reactivity product as the exhaust-hydrocarbon mixture). After elimination of interference due to quenching (by H_2O) of the chemiluminescence and optimisation of instrumental parameters, the ethylene-equivalent concn. provided by the analyser for some 55 compounds correlate to within 5% with those measured by g.c. P. C. R.

1H13. **Personal vinyl chloride monitoring device with permeation technique for sampling.** Nelms, Leonard H.; Reiszner, Kenneth D.; and West, Phillip W. (Environ. Sci. Inst., State Univ., Baton Rouge, La. 70803, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 994–998.—The device consists of a small tube containing a dimethylsilicone-rubber membrane and activated charcoal; the vinyl chloride (**I**) permeates the membrane and is adsorbed by the charcoal. Subsequently, **I** is desorbed by heating the tube to 300° and swept by a stream of He to a g.c. column (2 m × 2·4 mm) packed with Chromosorb 102 and operated at 90° with flame ionisation detection. The coeff. of variation at the level of 1 p.p.b. of **I** is 50%, while that at levels >0·1 p.p.m. is 5%.
G. P. C.

1H14. **Gas-chromatographic determination of acetic acid in industrial atmosphere and waste water.** Esposito, George E.; and Schaefer, Karen K. A. (U.S. Army Environ. Hyg. Agency, Aberdeen Proving Ground, Md. 21010, U.S.A.). *Am. Ind. Hyg. Assoc. J.*, 1976, **37** (5), 268–273.—*Procedure for air.* Air (20 l) was drawn (1 to 2 l min^{-1}) through two bubblers, each containing H_2O (20 ml) and 0·1N-Na_2CO_3 (2 ml). The bubbler contents were mixed and propionic acid was added as internal standard (0·5 ml of a 1 μg μl^{-1} soln.), the soln. was then evaporated and the residue dried at 110°. The acids were regenerated by addition of 0·5N-HCl (1 ml). *Procedure for water containing* 0·01 *to* 10 *p.p.m. of acetic acid.* Waste water (25 ml) was mixed with 0·5 ml of the propionic acid soln. and adjusted to pH 10 with N-Na_2CO_3, the soln. was then evaporated, dried at 110° and the organic acids were regenerated by addition of 6N-HCl (to pH 2). *Procedure for water containing* 10 *to* 200 *p.p.m. of acetic acid.* A 2-ml sample was mixed with 1 ml of the propionic acid soln. and adjusted to pH 2 with 6N-HCl. For g.l.c., each treated soln. was injected into a stainless-steel column (6 ft. × $\frac{1}{8}$ in.) packed with 4% of 85% H_3PO_4 on Porapak Q (120 to 150 mesh) operated at 190°, with He (35 ml min^{-1}) as carrier gas and flame ionisation detection. Before each injection 40% formic acid (3 μl) was injected to deactivate the column, and after 5 min the sample (3 μl) was injected together with 40% formic acid (1 μl). Sharp, symmetrical peaks were obtained and a graph of the area under the acetic acid peak *vs.* concn. was rectilinear for 250 to 1000 μg.
A. B. K.-S.

1H15. **Determination of dimethylnitrosamine in air and water by thermal-energy analysis: validation of analytical procedures.** Fine, David H.; Rounbehler, David P.; Sawicki, Eugene; and Krost, Ken (Thermo Electron Res. Center, Waltham, Mass. 02154, U.S.A.). *Environ. Sci. Technol.*, 1977, **11** (6), 577–580.—Methods involving use of a thermal energy analyser (TEA) (*cf.* Fine *et al.*, *Anal. Abstr.*, 1975, **29**, 5F14) are described for determining dimethylnitrosamine (**I**) in concn. down to 1 ng m^{-3} in air and 2 ng l^{-1} in water. The TEA is used as a detector with a gas chromatograph equipped with a stainless-steel column (6·5 m × 2 mm) packed with 15% of FFAP on AW DMCS Chromosorb W (80 to 100 mesh); the carrier gas is Ar (10 to 30 ml min^{-1}). A TEA - h.p.l.c. system incorporating a column (30 cm × 4 mm) of μPorasil, with 2,2,4-trimethylpentane - acetone (19 : 1) as mobile phase (2 ml min^{-1}), is also described. In control tests, no evidence was found for the formation of **I** during collection or analysis, but care must be taken when an appreciable concn. of O_3 is present. P. C. R.

1H16. **Determination of organomercurials as atmospheric pollutants and as preservatives of pharmaceutical and cosmetic products, even in the presence**

of metallic or ionic mercury. Gagliardi, L.; Amato, A.; Ricciardi, G.; and Chiavarelli, S. (Lab. Chim. Terap., Ist. Sup. Sanita, Rome, Italy). *Farmaco, Ed. Prat.*, 1977, **32** (6), 306–311 (in Italian).—Mercury derivatives, as their dithizone complexes, are separated by t.l.c., with hexane - acetone (9 : 1) as the mobile phase, extracted from the layer with dimethyl sulphoxide - $CHCl_3$ (3 : 1), and determined spectrophotometrically at the optimum wavelength for the particular compound (between 492 and 522 nm). For arylmercury compounds, sample preparation involves solubilising with 5% $Na_2B_4O_7$ soln. followed by extraction with $CHCl_3$ after acidification to pH 5 with acetic acid; alkylmercury compounds are extracted with benzene after acidification with HCl (*cf.* Westoo, *Anal. Abstr.*, 1968, **15**, 6942). Amounts down to from 5 to 20 μg of individual compounds can be determined with 97% recovery.
E. C. A.

1H17. **Gas-chromatographic determination of [traces of] styrene as its dibromide.** Hoshika, Yasuyuki (Environ. Res. Centre, Nagoya-shi, Aichi, Japan). *J. Chromatogr.*, 1977, **136** (1), 95–103.—The soln. of styrene in hexane (25 ml) is treated with aq. Br (2 to 400 μl) to give at least a 20 : 1 Br-to-styrene ratio, and heated to 40° for 5 min, and the excess of Br is removed in a stream of N. The soln. is subjected to g.l.c. on a glass column (1 m × 3 mm) packed with 3% of OV-17 on Chromosorb W (60 to 80 mesh) and operated at 250° with N as carrier gas (55 ml min^{-1}) and an electron-capture detector. The limit of detection was 0·01 ng, and of 138 compounds tested for interference dodec-1-ene and *o*- and *m*-cresol had the greatest effect, but the cresols could be removed by reaction with dil. aq. alkali. The method was applied to the determination of styrene in urban air.
M. J. St.

1H18. **Fluorimetric method of analysis for atmospheric benzo[α]pyrene.** Jager, J. (Inst. Hyg. and Epidemiol., Prague, Czechoslovakia). *Fresenius' Z. Anal. Chem.*, 1977, **284** (4), 283–285.—Benzo[*a*]-pyrene (**I**) in air-borne particulate matter is determined by measurement of its fluorescence in conc. H_2SO_4 soln. at 540 nm (with excitation at 522 nm). The glass-fibre filters are heated *in vacuo* at 315° for 45 min, and a soln. of the sublimate is submitted to t.l.c. on silica gel to separate **I** from other polycyclic aromatic hydrocarbons; the mobile phase is heptane - benzene (4 : 1). The zone containing **I** (located by comparison, in u.v. radiation, with authentic **I** on the same chromatogram) is extracted with cyclohexane plus a little dimethylformamide, the extract (≃9 ml) is shaken for 1 min with 3 ml of H_2SO_4, and the fluorescence of the acid layer is referred to a calibration graph (rectilinear for up to 1 μg of **I** in the 3 ml of acid). The lowest detectable amount of **I** is 5 ng, and the fluorescence is stable for at least 15 min with 90 to 4000 ng of **I**. A standard-addition method can also be used. Possible interference from incompletely separated fluorescence quenchers is discussed.
C. K. L.

1H19. **Characterisation of aza-arenes in basic organic portion of suspended particulate matter.** Dong, Michael W.; Locke, David C.; and Hoffmann, Dietrich (Dept. Chem., CUNY, Graduate Div., Queens College, Flushing, N.Y. 11367, U.S.A.). *Environ. Sci. Technol.*, 1977, **11** (6), 612–618.—Particulate matter collected on glass-fibre filters is extracted with benzene - methanol (4 : 1) in a Soxhlet apparatus, and the basic fraction derived from the extract by partition against H_2SO_4 is subjected to h.p.l.c. on LiChrosorb SI-60 (silanised). The mobile phase (initially a 0·5% soln. of isopropyl alcohol in hexane) is linearly programmed to 20% of isopropyl alcohol in $CHCl_3$ in 20 min (15 min after injection), and 8 sub-fractions are collected, concentrated, dissolved in methanol, and submitted to g.l.c. - m.s. or reversed-phase h.p.l.c. with identification by u.v. spectrophotometry or fluorimetry. Over 20 aza-arenes and nitrogenous bases have been identified, and quantitative results are presented for these compounds.
P. C. R.

1H20. **Determination of the biocide 1,3,5-triethylhexahydro-1,3,5-triazine in the atmosphere.** Tolan, Doina; Kalmutchi, George; and Tolan, Mircea. *Rev. Chim. (Bucharest)*, 1977, **28** (5), 475–476 (in Romanian).—Draw the air at 300 ml min^{-1} for 30 min through two absorbers, each containing 2 ml of 0·5% H_2SO_4, then heat the combined absorbent soln. at 90° for 10 min to convert the biocide (**I**) into formaldehyde and triethylamine. Cool, add an equal vol. of 0·5% disodium chromotropate in conc. H_2SO_4, add conc. H_2SO_4 to give a final concn. of 86% H_2SO_4, and, after 15 min, measure the absorbance at 570 nm to determine the formaldehyde (and hence the **I**). Beer's law is obeyed for the equiv. of 0·1 to 2·5 μg ml^{-1} of **I** in the final soln. The relative errors in determining 1, 1·5 and 2·5 μg ml^{-1} of **I** were 4·6, 2·7 and 2·5%, respectively.
C. D. K.

1H21. **Sampling, preservation and storage of water samples for analysis of metals.** Henriksen, Arne; and Balmer, Karin (Inst. Wat. Res., Oslo, Norway). *Vatten*, 1977, **33** (1), 33–38.—Procedures are described that minimise contamination of the sample by heavy metals during storage. All equipment is washed before use as follows—soak in a soln. of 1% of Na_2edta and 5% of Deconex detergent (Borer Chemie, Zuerich) for 24 h, rinse in glass-distilled H_2O, soak in 0·1N-HNO_3 for 1 h, and rinse thrice with glass-distilled H_2O. Samples are taken in polyethylene bottles, stored in 25-ml glass phials with polyethylene snap-on caps, and preserved by the addition of 0·25 ml of 3·5N-HNO_3. Statistical analysis shows that variance due to the glass phials is of the same order of magnitude as random errors.
R. J. F.

1H22. **2-Mercaptobenzothiazole supported on silica gel for chromatographic concentration of cadmium, copper, lead and zinc in natural water samples.** Terada, Kikuo; Inoue, Akihiko; Inamura, Junko; and Kiba, Toshiyasu (Dept. Chem., Fac. Sci., Kanazawa Univ., Ishikawa, Japan). *Bull. Chem. Soc. Jpn.*, 1977, **50** (5), 1060–1065.—Silica gel (60 to 80 mesh) was treated with a soln. of 2-mercaptobenzothiazole in dioxan - methyl acetate (4 : 1), then dried, washed with H_2O and again dried. The product (10 g) was packed into a column (7 cm × 2 cm) through which the sample (adjusted to pH 7) was passed at 2 to 5 l h^{-1}. Zinc was eluted with acetate - HCl buffer soln. of pH 4 and Cu plus Pb with acetone - HCl (9 : 1). From another portion of sample on another column, Cd and Zn were eluted with 1% HCl and Cu with acetone - HCl (9 : 1). All the metals were determined by a.a.s. The technique was applied to saline and non-saline waters. Interference by various metal ions, edta and citric and tartaric acids is discussed.
R. P. E.

1H23. **Determination of different forms of metal ions in natural water.** Linnik, P. N.; and Nabivanets, B. I. *Gidrobiol. Zh.*, 1977, **13** (1), 103–111 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G228.

1H24. **Application of anodic-stripping voltammetry to determination of the state of complexation of traces of metal ions at low concentration levels.** Branica, M.; Novak, D. M.; and Bubic, S. (Lab. Phys.-Chem. Sep., Centre Marine Res., Ruder Boskovic Inst., Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1977, **49** (3), 539–547.—A modified method involving stripping voltammetry at a hanging-mercury-drop electrode is described for the determination of metal ions in water at concn. down to 0·1μM; the reference and counter electrodes used are, respectively, a s.c.e. and a platinum wire. The sensitivity of the method and the magnitude of the critical apparent potentials for Cd and Pb are compared with the limits of detection and $E_{\frac{1}{4}}$ values obtained for the metals in d.c. polarography. The change in the critical potential as a function of the concn. of different anions was investigated as a means of determining the extent of complex formation by heavy metals in soln. The value of the critical potential was independent of the concn. of the metal ion but varied with that of the complexing agent.
B. D. H.

1H25. **Neutron-activation analysis of marine sediments.** Damburga, N.; Strauss, I.; and Mikelsone, G. *Latv. PSR Zinat. Akad. Vestis, Fiz. Teh. Zinat. Ser.*, 1977, (1), 7–13 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G232.

1H26. **Determination of trace elements in river water by neutron-activation analysis.** Nagatsuka, Sumiko; and Tanizaki, Yoshiyuki (Metrop. Isotope Res. Centre, Setagaya-ku, Tokyo, Japan). *Radioisotopes*, 1976, **25** (7), 375–379.—Samples were preconcentrated by evaporation to dryness; alternatively, volatile elements were co-pptd. with $Fe(OH)_3$, $Al(OH)_3$ or PbS (*Ibid.*, 1974, **23**, 693). Irradiation was carried out for 1 to 5 or 10 to 12 h in a thermal-neutron flux of $\simeq 2 \times 10^{12}$ n cm^{-2} s^{-1}. After an appropriate decay period, activities were measured with a Ge(Li) detector. The elements determined were Al, Ca, Dy, Mg, Ti and V (by brief irradiation), as well as Ag, As, Co, Cr, Cs, Cu, Eu, Fe, La, Lu, Na, Rb, Sb, Sc, Se, Sm, W and Zn (by prolonged irradiation); contamination of the elements at the pre-concentration stage appeared to be slight.
E. G. B.

1H27. **Determination of sodium, chlorine, iodine and sulphur in mineral water by neutron-activation analysis and X-ray fluorescence.** Al-Jobori, S. M.; Szegedi, S.; and Pazsit, A. (Inst. Exp. Phys., L. Kossuth Univ., Debrecen, Hungary). *Radiochem. Radioanal. Lett.*, 1977, **30** (1), 45–51.—Samples were irradiated (^{252}Cf source in a water moderator) for 2 h (for Na) or 1 h (for Cl and I), and, after 2 min, the activities at 1368 keV (^{24}Na), 1642 keV (^{38}Cl) and 443 keV (^{128}I) were measured for 1 h with a Ge(Li) detector. The sensitivity was 10 p.p.m. for each of these elements. To determine S, X-ray fluorescence (^{55}Fe source) was used, with measurement of the 6·4-keV $K\alpha$ peak [Si(Li) detector]; the sensitivity was $\simeq$30 p.p.m., and there was no interference from Na or Cl.
J. W. P.

1H28. **Two specialised delayed-neutron detector designs for assays of fissionable elements in water and sediment samples.** Balestrini, S. J.; Balagna, J. P.; and Menlove, H. O. (Los Alamos Sci. Lab., Univ. Calif., N.Mex. 87544, U.S.A.). *Nucl. Instrum. Meth.*, 1976, **136** (3), 521–524.—The detectors described are used for rapid assays of fissionable elements by sensing for delayed neutrons emitted by samples after irradiation in a nuclear reactor. The more sensitive of the detectors, designed to assay for U in water samples, is 40% efficient; the other, designed for assays on sediment samples is 27% efficient. These detectors are also designed to operate under water as a means of shielding against neutron leakage from the reactor and neutrons from cosmic rays.
R. J. F.

1H29. **Trace determination of copper in steam-generator-feed waters and condensates by the ion-exchange-enrichment method.** Hellwig, K. *Chem. Tech. (Leipzig)*, 1977, **29** (6), 329–331 (in German).—The Cu is enriched by adsorption on a Wofatit MC-50 ion-exchange column; breakthrough curves are presented for Fe^{2+} and Cu^{2+} at pH 4·6, and for Cu^{2+} at pH 1·7 and 3·2. The eluted Cu^{2+} are determined photometrically (with tetraethylthiuram disulphide) at 425 nm; the coeff. of variation ranges from 4 to 28% for 5 to 0·5 μg l^{-1} of Cu.
J. K. C.

1H30. **Effect of surfactants on determination of aluminium in waters.** Pakalns, P.; and Farrar, Yvonne J. (Australian Atom. Energy Comm. Res. Establ., Lucas Heights, N.S.W.). *Water Res.*, 1977, **11** (4), 387–392.—In a study of the interfering effects of 11 surfactants on the spectrophotometric determination of Al (16 to 160 μg dl^{-1}) by the Eriochrome cyanine R (C.I. Mordant Blue 3), ferron - 1,10-phenanthroline and Chrome Azurol S (C. I. Mordant Blue 29) methods, results showed that cationic, anionic and non-ionic detergents at concn. up to 2, 100 and 1000 mg l^{-1}, respectively, can be tolerated. Sodium triphosphate, however, interferes at concn. >1 mg l^{-1}, and nitrilotriacetic acid, a commercial soap and $Na_4P_2O_7$ have to be at levels of <0·5, <10 and <20 mg l^{-1}, respectively, to limit errors to 5%. Of the three methods cited, that based on ferron - 1,10-phenanthroline permits the largest concn. of surfactants to be tolerated, provided that no Fe is present.
P. C. R.

1H31. **Determination of indium content of saline water in hydrogeological tracer investigations by activation with a californium-252 neutron source.** Lebecka, J. (Chief Inst. Mining, Katowice, Poland). *Rap., Inst. Fiz. Jad., Krakow*, INT-94/I, 1976. Pp. 55–62 (in Polish).—Indium (the edta complex of which is used as a tracer) is determined by activation of the water sample with thermal neutrons from a ^{252}Cf source and measurement of the 1293-keV line of ^{116}In. For water of total salinity <1 g l^{-1}, the sample (5 l), in a cylindrical vessel, is irradiated directly, and the In - edta complex is collected on Amberlite IRA-400 resin for subsequent activity measurement. The sensitivity with a 1-mg ^{252}Cf source is 2·5 ng g^{-1}. At total salinities from 1 to 50 g l^{-1}, the In is co-pptd. with at least a 10-fold excess of Fe^{III} by adding NaOH, and the mixed hydroxides are collected for irradiation and subsequent measurement; the sensitivity (1-mg source) is 0·8 ng g^{-1}. There is no interference from Br or I; interference from ^{24}Na (at 1368 keV) is avoided by the use of a sensitive semiconductor detector.
P. B.

1H32. **Combined ion-exchange paper and X-ray fluorescence method for determination of uranium in natural waters.** Minkkinen, Pentti (Helsinki Univ. Technol., Dept. Chem., Espoo, Finland). *Finn. Chem. Lett.*, 1977, (4–5), 134–137.—A 100-ml sample of water (adjusted to pH 2·5 with HCl) is filtered (at 15 ml min^{-1}) five times through a 19-mm disc of Whatman P-81 cellulose phosphate paper (NH_4^+

form). After drying, the disc is subjected, *in vacuo*, to X-ray excitation with use of a tungsten-target tube, and the intensity of the U $L\alpha$ line and of the background are measured with use of a LiF(200) crystal and a scintillation counter. The response is rectilinear for up to 1 mg of U, the sensitivity is 10 c.p.s. μg^{-1} of U, and the detection limit is 0·3 μg for a counting time of 100 s. P. C. R.

1H33. **Direct method for measuring inorganic and organic carbon in recent sediments.** Kotsch, Richard W.; D'Itri, F. M.; and Upchurch, Sam B. (Dept. Geol., State Univ., East Lansing, Mich. 48824, U.S.A.). *J. Sediment. Petrol.*, 1976, **46** (4), 1026–1030.—The sample is treated with H_2SO_4 - H_3PO_4 to decompose carbonates into CO_2, then organic C is converted into CO_2 by treatment with chromic acid - HIO_3 and heating to 210°. The gases evolved are swept by a stream of CO_2-free air through absorption vessels to remove HCl, H_2S and H_2O, then CO_2 is absorbed by ascarite - $Mg(ClO_4)_2$, and the increase in wt. of the absorbent is measured. The technique is applicable to a wide range of clay- and carbonate-rich glacial and lacustrine sediments. P. C. R.

1H34. **Determination of total cyanide: scope and field of application.** Analytical Chemists' Committee of Imperial Chemical Industries Ltd. *Effluent Water Treat. J.*, 1977, **17** (2), 77–79.—The method is similar to that previously described (*Anal. Abstr.*, 1977, **32**, 2H45) and is applied to natural waters and effluents. The sample, mixed with Pb acetate soln., is boiled under reflux (apparatus shown in a diagram) with CuCl soln. in HCl, and the HCN liberated is swept by a stream of air into aq. NaOH. After passage of the gases for 1 h, the alkaline soln. is acidified with acetic acid, then chloramine T and pyridine - pyrazolone reagents are added, the mixture is kept in the dark for 30 min and diluted with H_2O, and the absorbance of this soln. is measured at 620 nm. The detection limit is 0·01 p.p.m.; aldehydes and ketones interfere. R. P. E.

1H35. **Analysis of nitrogen forms in waters and waste waters.** Jenkins, D. *Prog. Water Technol.*, 1977, **8** (4–5), 31–53.—A review is presented on current practice in analysis for organic N, NH_3, NO_2^- and NO_3^- over various concn. ranges in natural waters, with particular reference to the precision of the different methods. (51 references.)

WRC Inf.

1H36. **Aquatic nitrogen determinations in real-time.** Helfgott, T.; and Mazurek, J. S. *Prog. Water Technol.*, 1977, **8** (4–5), 433–446.—Details are given of techniques for rapid determination of N compounds at various points in a sewage-treatment plant, using a NO_3^- probe, an NH_3 probe, and a pyrolysis - microcoulometric analyser for total N. Total N can be determined within 15 min, and NO_3^- and NH_3 within 1 min; organic N is determined by subtracting the amounts of NH_3 and NO_3^- from that of total N. The feasibility of using these techniques for regular monitoring is discussed.

WRC Inf.

1H37. **Analysis of ammonia in polluted sea-water.** Hampson, B. L. (Min. Agric., Fish. and Food, Fish. Lab., Lowestoft, Suffolk, England). *Water Res.*, 1977, **11** (3), 305–308.—The sample (50 ml) is passed through a glass-fibre filter-paper, and to the filtrate are added 2 ml of ethanolic 10% phenol, 5 ml of chlorinating agent [200 mg of 1,3-dichloro-6-hydroxy-1,3,5-triazine-2,4-dione (Na salt), 3·2 g of NaOH and 40 g of Na citrate in 100 ml of H_2O] and 2 ml of aq. 0·5% $K_4Fe(CN)_6$. The mixture (maintained at 30° ± 1°) is exposed to 365-nm radiation for at least 40 min to develop the indophenol blue complex, and the absorbance of the resulting soln. is measured at 640 nm. Solutions of $(NH_4)_2SO_4$ are used as the external standard, and known additions of NO_2^- are made to the test soln.; use of this procedure avoids interference from amino-acids, amines, NO_2^- and H_2S. The limit of detection is 2 p.p.b. of ammonia-N in sea-water. P. C. R.

1H38. **Determination of arsenic in water, rocks and sediments by atomic-absorption spectrophotometry.** Guimont, J.; Pichette, M.; and Rheaume, N. (Anal. and Control Service, Mineral Res. Centre, Dept. Natural Resources, Ste-Foy, Quebec, Canada). *At. Absorpt. Newsl.*, 1977, **16** (3), 53–57.—Arsenic was determined by reduction with $NaBH_4$ and a.a.s. at 193·7 nm in a N - H flame. A deuterium background corrector and a Perkin-Elmer hydride generation system were used, with an electrodeless discharge lamp as light source. Concentrate, mineral and rock samples (0·5 g) were decomposed by fusion with NaOH and dissolved to a vol. of 50 ml with H_2O. Sediment and soil samples (0·5 g) were heated overnight at 70° with HNO_3, then were heated to dryness, and each residue was leached with 3·6M-HCl (15 ml). Portions of sample soln. (20 ml for water samples, 10 ml for concentrate, mineral or rock samples, 5 ml for sediment or soil samples) were treated with aq. 5% KSCN (1,1 or 2 ml, respectively) and made acid with HCl, and a pellet of $NaBH_4$ ($\simeq$0·25 g) was added. The AsH_3 produced was aspirated into the flame by use of the hydride generation system. The method was rapid (125 samples per man-day), and had an absolute detection limit of 18 ng. The coeff. of variation at the 25-μg l^{-1} level in water and the 5-p.p.m. level in rocks and sediments were <7%. Practical detection limits were 1 μg l^{-1}, 0·2 p.p.m. and 0·1 p.p.m. for water, rocks and sediments, respectively. The effects of various foreign elements were investigated at concn. equal to or greater than those found in the samples to be analysed; only Ni interfered significantly, this effect being minimised by the addition of the KSCN. Analysis of standard rock samples, and standard additions of As to samples, showed that the method gave accurate results. C. K. L.

1H39. **Molecular spectral interference in the determination of arsenic by furnace atomic absorption.** Poldoski, J. E. (U.S. Environ. Prot. Ag., Duluth, Minn. 55804). *At. Absorpt. Newsl.*, 1977, **16** (3), 70–73.—Such interference, which could cause large errors in the determination of As in natural water by graphite-furnace a.a.s., was found to be associated with certain inorganic materials, especially silicate minerals, in the sample. The interference, which could cause measured As concn. to be erroneously high by a factor of five, was eliminated by digestion under pressure with HF, but it could also be reduced by addition of HF to the undigested sample before injection into the furnace. A bandpass of 0·2 nm should be used. C. K. L.

1H40. **Automatic determination of sulphate in water samples and soil extracts containing large amounts of humic compounds.** Ogner, G.; and Haugen, A. (Forest Res. Inst., As, Norway). *Analyst* (*London*), 1977, **102** (1215), 453–457.—Water samples were preserved by means of HCl

(7 ml per litre of sample) and stored at 4°; soil extracts were prepared by shaking the air-dried sample for 1 h with 0·01M-$Ca(H_2PO_4)_2$, preserved with $CHCl_3$. The treated samples were dialysed, the diffusates were mixed with a supplementary SO_4^{2-} soln. (10 mg l^{-1} of sulphate-S) and $BaCl_2$ - Tween 80 soln. and the turbidity of each was measured with a nephelometer. Samples (24 per h) containing 1 to 100 mg l^{-1} of sulphate-S could be analysed. R. W.

1H41. **[Detection of] residual chlorine in drinking water.** Thorsell, W.; Malm, E.; and Mikiver, M. (Natl. Defence Res. Inst., Stockholm, Sweden). *J. Am. Water Works Assoc.*, 1977, **69** (4), 209–210.—A field test is described, which does not involve the use of hazardous reagents. Equal parts by wt. of KI, K H tartrate and sol. starch (stored as either a powder or tablet sealed in foil) are added to the sample to produce a blue starch - iodine complex, which indicates the presence of Cl. WRC Inf.

1H42. **Solid-state exchange colorimetry in successive analysis of iron(II) and iron(III) in solution.** Gadia, M. K.; and Mehra, M. C. (Environ. Contaminants Res. Group, Chem. Dept., Univ. Moncton, N.B., Canada). *Microchem. J.*, 1977, **22** (1), 27–33.—A soln. containing Fe (up to 20 p.p.m.) was adjusted to pH 2 with HNO_3 and diluted to 50 ml. After adding $Hg[Co(SCN)_4]$ (400 to 500 mg), the mixture was stirred for several minutes then set aside for 1 h. Tervalent Fe gave a red-coloured soln., the absorbance of which was measured at 460 nm; Beer's law was obeyed for 0·1 to 8 p.p.m. of Fe. Another aliquot of the soln. was treated in a similar way, but with the addition of H_2O_2; this oxidised Fe^{II}, and total Fe was determined as Fe^{III}. There was no interference from a 10 to 50-fold excess of, *e.g.*, Ca, Co, Cd, Mn, Na, Cl^-, NO_3^-, SO_4^{2-} and acetate, but Hg interfered at all concn. and Cu and Zn present together showed some interference. Results for tap-, lake- and river-water agreed well with those obtained by use of 1,10-phenanthroline. T. G. A.

1H43. **Gas-stripping, sorption and thermal-desorption procedures for pre-concentrating volatile polar water-soluble organics from water samples for analysis by gas chromatography.** Kuo, P. P. K.; Chian, E. S. K.; DeWalle, F. B.; and Kim, J. H. (Environ. Eng., Civil Eng. Dept., Univ. Illinois, Urbana, Ill. 61801, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1023–1029.—The efficiency of removal of organic compounds from aq. soln. (*e.g.*, drinking water) by stripping with He, the adsorption of the stripped compounds on Tenax GC (35 to 60 mesh) and their subsequent thermal desorption were studied, with use of, *e.g.*, alcohols, aldehydes and ketones as the principal model compounds. With use of the apparatus described by Bellar and Lichtenberg (*Anal. Abstr.*, 1975, **29**, 6H33), the adsorption and desorption efficiencies were >75 and 80%, respectively, for the majority of the compounds studied. Break-through and displacement of lower alcohols occurs on Tenax GC under the experimental conditions used. G. P. C.

1H44. **Some conditions for use of macro-recticular resins in quantitative analysis of organic pollutants in water.** Stepan, S. F.; and Smith, J. F. (Dept. Phys. Chem., La Trobe Univ., Bundoora, Melbourne, Vic., Australia). *Water Res.*, 1977, **11** (4), 339–342.—Separation of the pollutants (10-p.p.m. level) from the water sample was effected on columns packed with 2 g of XAD-2 (a non-polar polystyrene - divinylbenzene copolymer) or of XAD-7 (a polar polymethacrylate polymer), with a sample flow rate of up to 30 ml min^{-1} and ethyl ether as eluent. Use of the XAD-2 column was supplemented by a head-space extraction procedure to remove most of the volatile material; for the XAD-7 column, the sample was adjusted to pH 1·0 to 1·5. Each eluate obtained was analysed by g.l.c. on a column of 3% of SE-30 (for non-polar compounds) or of 8% of di-isodecyl phthalate - H_3PO_4 (3:5) (for polar compounds) on DMCS Chromosorb G, with flame ionisation and (in some instances) m.s. detection. For the XAD-2 column, average recoveries (5 results) ranged from 27% (phenol) to 90% (naphthalene); the corresponding values for the XAD-7 column were 45 and 93%. P. C. R.

1H45. **Measurement of total organic carbon (TOC) in water at lower levels.** Reijnders, H. F. R.; Roemer, F. G.; and Griepink, B. F. A. (Natl. Inst. Public Health, Bilthoven, Netherlands). *Fresenius' Z. Anal. Chem.*, 1977, **285** (1), 21–23.—Doubt is expressed as to the validity of TOC values below 10 p.p.m. Specific sources of error discussed include the absence of suitable standards, the value of the natural CO_2 blank, the combustion mechanism, questionable extrapolation of calibration graphs, the application of combustion catalysts, and output-signal peak-height measurement. J. K. C.

1H46. **Measurement of stable-isotope ratio of organic carbon in water samples.** Fujii, Toshihiro; and Otsuki, Akira (Natl. Inst. Environ. Studies, Tateno, Yatabe-machi, Tsukuba-gun, Ibaraki, Japan). *Bunseki Kagaku*, 1977, **26** (6), 361–364 (in Japanese).—The method was developed using samples from an urban river. After wet oxidation of organic C to CO_2 in a sealed ampoule, the isotope ratios were determined by g.c. - m.s. in combination with a total organic-C analyser. Helium was used as carrier gas and silica gel (60 to 80 mesh) as the column packing for g.c. A multiple-ion detector - digital integrator system measured intensities at *m/e* 44 and 45 at a switching rate of 1 ms. Measurements of CO_2 from 53 μg of Na_2CO_3 gave isotope ratios with a coeff. of variation of 0·62%, but those obtained for organic C in natural waters had coeff. of variation 2 to 3 times higher.

1H47. **Determination of chlorinated hydrocarbons in drinking water.** Cavallaro, Aldo; and Grassi, Piero (Lab. Chim. Prov., Milano, Italy). *Boll. Lab. Chim. Prov.*, 1976, **27** (11), 337–350 (in Italian).—Two g.l.c. methods are described. In method (*i*), the sample (200 ml) was extracted with 25 ml of ethyl ether or light petroleum, and 2 μl of the extract was injected directly on to the column (of SE-30, XE-60 or Carbowax 400). In method (*ii*), the sample (up to 500 μl) was diluted (if necessary) to 500 μl with H_2O, and the soln., mixed with $MgSO_4.7H_2O$ (0·7 g in 2 ml of H_2O), was set aside for ≃30 min; 100 μl of the head-space vapour was then transferred to a glass column (3 m × 3·5 mm) packed with 5% of squalane on Chromosorb W (60 to 80 mesh) and operated at 60°, with Ar - CH_4 (9:1) as carrier gas and electron-capture detection. The compounds determined (at p.p.b. levels) were $CHCl_3$, CCl_4, trichloroethane and tri- and tetrachloroethylene; recoveries were 100% in each instance except for $CHCl_3$ (90%). Automated sampling techniques could be used, and it was possible to analyse 36 samples in 8 h. L. B

1H48. **Sample-bottle purging method for the determination of vinyl chloride in water at sub-microgram-per-litre levels.** Dressman, Ronald C.;

and McFarren, Earl F. (Wat. Supply Res. Div., Municipal and Environ. Res. Lab., U.S. Environ. Protect. Agency, Cincinnati, Ohio 45268, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (2), 69–72.—Vinyl chloride is transferred from the aq. phase to the gaseous phase by the procedure of Bellar and Lichtenberg (*Anal. Abstr.*, 1975, **29**, 6H33), except that the 40-ml sample bottle is purged directly. In place of a column of Chromosorb 101, a glass column (180 cm × 4 mm) packed with Chromosorb 101 (for 60 mm) and Chromosorb 102 (for 120 mm) is used to eliminate interference by CNCl. The column is temp.-programmed from 50° to 160° at 10° min^{-1}, with N as carrier gas (80 ml min^{-1}) and a detector consisting of a Dohrman micro-coulometric titration system. The method can be used to detect 100 ng l^{-1} of vinyl chloride in water.

M. J. Sa.

1H49. **Determination of hydrocarbons in waters.** Ranchet, J.; and Clement, P. (Lab. Reg. Ponts et Chausses Ouest Parisien, France). *Tech. Sci. Munic. Eau*, 1976, **71** (10), 23–27 (in French).—The basic method described (API 733–58), which is suitable for effluents containing hydrocarbons only, involves extraction by CCl_4, i.r. spectroscopy of the extract at 2924 cm^{-1}, and determination of the hydrocarbons by reference to a calibration graph. If fatty acids also are present, the absorbance is measured at 1710 cm^{-1} and 2924 cm^{-1}; fatty acids are determined at 1710 cm^{-1}, with use of a calibration graph for a standard fatty acid mixture. The corresponding absorbance at 2924 cm^{-1}, obtained from the graph, is subtracted from the total absorbance to give a corrected value for the hydrocarbons alone. If fatty acids and esters are present, the CCl_4 extract is subjected to chromatography on a column of Florisil before spectroscopy at 2924 cm^{-1}. The limit of detection for hydrocarbons in water is 0·1 mg l^{-1}; the best precision is achieved by use of the appropriate hydrocarbons or contaminants in preparing the calibration graphs. R. J. F.

1H50. **Application of glass-capillary columns to monitor petroleum-type hydrocarbons in marine sediments.** Overton, E. B.; Bracken, J.; and Laseter, J. L. (Dept. Biol. Sci., Univ., New Orleans, La. 70122, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (5), 169–176.—Samples are dried *in vacuo* at 40° and a portion (100 g) is extracted under reflux with hexane and benzene, each for 7 h. The combined extracts are reduced in vol. and transferred to a column containing 1 cm of activated alumina over 20 cm of activated silica gel. Aliphatic compounds are eluted with hexane and aromatic compounds with benzene. The two fractions are reduced in vol. and aliquots are injected on to a glass column (35 m × 0·3 mm), the surface of which has been modified by deposition of $BaCO_3$ and deactivation with successive layers of Carbowax 20M and Emulphor and finally coated with SE-52. The column is temp.-programmed at 2° min^{-1} from 90° to 240°, at which temp. it is held for 115 min. The column efficiency (1600 plates m^{-1}, measured at 154° with octadecane) is sufficient to separate almost all isoprenoid, branched and cyclic hydrocarbons associated with petroleum contamination, and also to distinguish aromatic contaminants from indigenous compounds. M. J. Sl.

1H51. **Detection and spectrophotometric determination of 126 phenolic compounds in water using four group-specific reagents.** Koppe, P.; Dietz, F.; Traud. J.; and Ruebelt, C. (Chem. u. Biol. Lab. Ruhrverband, Essen, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **285** (1), 1–19 (in German).—Details of methods for determining phenols with 4-nitroaniline, sulphanilic acid, 4-aminoantipyrine and 3-methylbenzothiazolin-2-one hydrazone are presented, and the spectrophotometric data for 126 compounds are listed. The effects of substituent type and substitution position on the value of the absorption coeff. of the resulting derivative are discussed. Analytical efficiencies of 56 to 74% are reported for the four reagents. J. K. C.

1H52. **Gas - liquid chromatographic method with electron-capture detection for determination of residues of some phenoxyacetic acid herbicides in water as their 2,2,2-trichloroethyl esters.** Mierzwa, S.; and Witek, S. (Inst. Org. and Polymer Technol., Tech. Univ. Wroclaw, Poland). *J. Chromatogr.*, 1977, **136** (1), 105–111.—Natural water (1 l) is acidified to pH 2 with H_2SO_4 and passed down a column (10 cm × 1 cm) containing Amberlite XAD-4 (6 cm deep) at 15 ml min^{-1}. The adsorbent is dried in a stream of N, the adsorbed phenoxyacetic acids are eluted with benzene (40 ml) at 5 ml min^{-1}, and the solvent is evaporated to dryness at 40° in a rotary evaporator. The residue is esterified by heating with 20% of 2,2,2-trichloroethanol in trifluoroacetic anhydride (1 ml) and conc. H_2SO_4 (5 μl) under reflux for 15 min. The reaction mixture is reduced to ≃0·1 ml in an air stream at room temp., 2,2,4-trimethylpentane (2 ml) and 0·5M-KOH (6 ml) are added, and the mixture is shaken. The organic layer (1 ml) is cleaned-up on a column (10 cm × 1 cm) containing silica gel (4 cm deep) and Na_2SO_4 (1 cm), which is then washed with 2,2,4-trimethylpentane (25 ml). The esters are eluted with benzene (15 ml), and the extract is reduced to 1 ml in a stream of N, and subjected to g.l.c. on a column (2 m × 3 mm) packed with 15% of QF-1 plus 10% of DC-200 on Chromosorb W AW-DMCS and operated at 195°, with N as carrier gas (80 ml min^{-1}) and an electron-capture detector. The recoveries (at 0·4 p.p.b.) were 92·1 ±3·4% for 2,4-D and 87·8 ± 2·1% for MCPA; the limits of detection for a 1-l water sample were 0·096 and 0·06 p.p.b., respectively. M. J. Sl.

1H53. **Occurrence and determination of a herbicide benthiocarb [*S*-(4-chlorobenzyl) diethylthiocarbamate] in rivers and agricultural drainages.** Suzuki, Manabu; Yamato, Yasuhiro; and Akiyama, Takashi (Munic. Inst. Environ. Health Sci., Kitakyushu, Japan). *Water Res.*, 1977, **11** (3), 275–279.—Benthiocarb (**I**) is extracted from water with hexane and is determined in the extract either by g.l.c. with a flame-photometric detector operated in the S mode (394-nm filter) or by use of a combined g.l.c. - m.s. - computer system. In each instance, the column (2 m × 3 mm) is packed with 2% of Apolar 10C on Gas-Chrom Q and is operated at 180° with N (40 ml min^{-1}) as carrier gas. The minimum detectable amount of **I** with flame-photometric detection is 0·56 ng and that by g.l.c. - m.s. is 100 ng. From 0·1 to 10 μg l^{-1} of **I** can be determined in river water.

P. C. R.

1H54. **Automatic potentiometric control of industrial waste waters.** Smolyakov, B. S.; and Velichko, V. N. (Inst. Inorg. Chem., Acad. Sci. USSR, Siberian Dept., Novosibirsk). *Zavod. Lab.*, 1977, **43** (6), 656–658 (in Russian).—The construction of an automatic potentiometric analyser is described. The measuring cell contains pH, pNa, and pCl ion-selective electrodes, and a membraneless thallium electrode for the determination of dissolved O. The last-named electrode is calibrated by the Winkler

method; the others are calibrated against standard soln. The accuracy and stability of the analyser, and the working life of the electrodes under continuous operation on cellulose factory effluents are discussed. A. S. W.

1H55. **Extractive spectrophotometric determination of mercury with thiobenzoylacetone [4-phenyl-4-thioxobutan-2-one]; analysis of waste water.** Murti, M. V. R.; and Khopkar, S. M. (Dept. Chem., Indian Inst. Technol., Bombay). *Bull. Chem. Soc. Jpn.*, 1977, **50** (3), 738–741.—The Hg^{II} was extracted from aq. soln. (25 ml) at pH 4·0 with a mM soln. of thiobenzoylacetone in benzene (10 ml). The separated organic phase was washed with $Na_2B_4O_7$ buffer of pH 12 to remove surplus reagent. The absorbance of the Hg complex was then measured at 345 nm against a reagent blank. Beer's law was obeyed in the range 0·6 to 12·1 $\mu g\ ml^{-1}$ of Hg. The effects of pH, reagent concn., and salting-out agents are discussed. The tolerance limits for 36 cations and 26 anions are listed; CN^- interfere. The application of the method to the analysis of an industrial effluent is described. T. G. A.

1H56. **Thermal controller for the short COD [chemical oxygen demand] determination.** Crandall, P. G.; Niementowski, C. C.; and Nelson, P. E. (Inst. Food and Agric. Sci., Univ. Florida, Lake Alfred, Fla. 33850, U.S.A.). *J. Food Sci.*, 1977, **42** (3), 839–842.—Apparatus is described by which waste water is heated with digesting chemicals in an Erlenmeyer flask under controlled conditions by heating over a Bunsen burner and shaking with a wrist-action shaker. The operation is controlled by a service head containing a glass rod depth-stop, thermometer and thermistor probe. As a result, the short COD test is improved in speed, safety and precision. The apparatus was used to analyse a glucose soln., and simulated tomato- and corn-processing wastes. The coeff. of variation for the test was 1·4% on glucose soln. and 2·7% on the tomato waste. R. F. S.

1H57. **Chromatographic determination of formaldehyde in waste waters.** Tkacheva, V. N.; Prokop'eva, M. F.; Galitskaya, N. B.; and Stebeneva, I. G. (Plastics Res. Inst., Moscow, USSR). *Zavod. Lab.*, 1977, **43** (6), 666–668 (in Russian).—The sample (≃0·1 ml) is slowly introduced into an evaporator (at 150°) connected to a chromatographic column (68 cm long) packed with Polysorb N or Porapak N. The temp. of the column is programmed from 60° to 140° at 6° min^{-1}, the carrier gas is He (45 ml min^{-1}), and detection is by thermal conductivity. Ethanol is used as internal standard. Separation of formaldehyde, water, ethanol and phenol is good, and formaldehyde can be determined in concn. down to 30 mg l^{-1}. A. S. W.

1H58. **Ultra-micro analysis of organic substances in waste water from a duprene [neoprene] plant.** *Acta Chim. Sinica*, 1976, **34** (2), 115–122 (in Chinese).—The method (used for monitoring the efficiency of a waste-water treatment plant) involved bubbling N (at 100 ml min^{-1}) through the sample (400 ml) for 30 min to sweep out the volatile organic substances (b.p. <140°), which were adsorbed on active carbon for subsequent g.l.c. (at 94°). The adsorbent was extracted with CS_2 (1 ml), and the extract was applied to a column packed with a 3:2 mixture of (*i*) 20% of di-iso-octyl phthalate on fire-brick 6201 (60 to 80 mesh) and (*ii*) 20% of tetra(cyanoethoxymethyl)methane on the same support; N was used as carrier gas and detection was by flame ionisation. Recoveries for chloroprene, hexa-1,5-dien-3-yne and acetaldehyde ranged from 97 to 100%, and their respective detection limits were 4, 4 and 6 p.p.b. (4 p.p.b. for benzene). S. H. Y.

1H59. **Direct-injection gas - liquid chromatographic analysis of xylenols in industrial waste water.** Baird, R. B.; Carmona, L. G.; and Jenkins, R. L. (San Jose Creek Water Quality Lab., Whittier, Calif. 90601, U.S.A.). *Bull. Environ. Contam. Toxicol.*, 1977, **17** (6), 764–767.—Samples were analysed on a glass column (2 m × 3 mm) packed with 4% of dinonyl phthalate on Chromosorb G (80 to 100 mesh) at 140°, with N as carrier gas (60 ml min^{-1}). Retention times, relative to phenol, are given for cresol and xylenol isomers. Typical analyses of petroleum-industry discharges are presented. S. M. M.

1H60. **NIOSH Manual of Analytical Methods.** Second Ed. by Taylor, David G. Publ.: U.S. Department of Health, Education, and Welfare, Cincinnati, Ohio, 1977. Vol. I: NIOSH Monitoring Methods. Pp. 790. $8.75. Vol. II and III: Standards Completion Program Validated Methods. Pp. 955; $9.75. Pp. 863; $9.00.

See also Abstracts: **1B27**, Determination of H_3BO_3 in reactor coolant water. **1B49**, **1B50**, Determination of Au in water. **1B61**, Determination of Sr in environmental samples rich in Ca. **1B65**, Determination of Zn in water. **1B70**, **1D15**, Determination of Cd in waste water. **1B73**, Extraction of Hg from, *e.g.*, environmental samples. **1B139**, Determination of NO in gases. **1B140**, Determination of NO_2^-. **1B141**, Determination of NO_3^- in water. **1B144**, Determination of N compounds in flue gas. **1C50**, Determination of traces of polycyclic aromatic hydrocarbons. **1C66** to **1C70**, Reviews on pollution associated with coal. **1C71**, Determination of CS_2 in coke-oven gas. **1C73**, Identification of dyes in polluted water. **1C80**, Determination of COD, BOD and TOC in waste materials associated with the paper industry. **1C90**, Determination of di-isocyanates in air: review. **1D7**, **1D8**, Analysis of tobacco smoke. **1D10**, Determination of Ca, Ba and Sr in water. **1F5**, **1F6**, Determination of Pb in air and water. **1G12**, Determination of anthracene in sediments. **1G13**, Determination of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin in environmental samples. **1G15**, Determination of heavy metals in sewage sludge. **1G24**, Determination of chloramphenicol in water. **1J5**, Determination of O in gases. **1J7**, Extraction of chemicals from water and sediments. **1J10**, Computerisation in automated analysis of water samples. **1J13**, Materials for sorption of metals from water. **1J16**, G.l.c. of pesticides in water. **1J111**, Determination of Cd, Cu, Pb and Zn in sea-water.

J—TECHNIQUES; APPARATUS

General

1J1. **Device for cutting capillary and heavy-wall glass tubing.** Frost, J. A. (Chem. Dept., Univ. Reading, Berks., England). *Lab. Pract.*, 1977, **26** (6), 481–483.—A frame (shown in a diagram) is fitted around the tube (previously scored at the

required position), and a light load is applied (at the cut-mark) by means of a screw. The glass snaps cleanly after ≃1 s. S. M. M.

1J2. **Evaporator for moisture-sensitive materials.** Conkerton, E. J.; Bourgeois, L. L.; and Ory, R. L. (S. Reg. Res. Center, U.S. Dept. Agric., New Orleans, La. 70179, U.S.A.). *Lab. Pract.*, 1977, **26** (6), 483, 481.—The heating block described for use with the Meyer N-Evap (Organomation Associates, Inc., Shrewsbury, Mass.) apparatus provides dry heating at higher temp. than are available with the standard water-bath heater (which it replaces). S. M. M.

1J3. **Plant sample segregation during grinding.** Lockman, R. B.; and Molloy, M. G. (Agron. Services Lab., AGRICO Chemical Co., Washington C.H., Ohio 43160, U.S.A.). *Commun. Soil Sci. Plant Anal.*, 1977, **8** (6), 437–440.—Dried maize ear-leaves were ground in a Wiley mill to pass a 40-mesh screen, and the ground material was collected sequentially in four sub-samples. Phosphorus, Ca, Mg, B, Cu, Fe, Mn, Al and Zn were determined in dry-ashed sub-samples by emission spectrography or a.a.s. Flame photometry, a turbidimetric - colorimetric procedure and an AutoAnalyzer were used for the determination of K, S and N, respectively, in wet-ashed sub-samples. Results for N, Ca, Fe and Al were found to be high for the first three fractions from the grinder, and the last fraction showed low results for N, P, K, Ca, S, Fe, Zn and Al. Results indicated that the analysis of a maize leaf would be over-estimated for most elements if the last portion of the ground sample was not mixed with the bulk of the material. E. K. O.

1J4. **Apparatus for determining moisture in gases.** Tulyakov, E. N.; and Rychkova, N. A. (State Inst. Appl. Chem., Perm Branch, USSR). *Zavod. Lab.*, 1977, **43** (6), 716–718 (in Russian).—The sample is passed through a 3-mm gap between a flat Perspex plate and a mirror, the back of which is fixed to the wall of a cooling chamber. The change in the reflectivity as the mirror is gradually cooled with liquid N is recorded with use of a photo-electric cell. The reflectivity changes abruptly at the dew-point, the corresponding temp. being determined with a suitably positioned thermocouple, and the moisture content is calculated from the dew-point tables. The method has been applied to industrial gases containing $>0{\cdot}3$ mg m^{-3} of moisture. A. S. W.

1J5. **New Draeger oxygen 5%/B tubes: determination of oxygen over the range 1 to 100% by volume.** Huneke, Karl Heinz. *Draegerheft*, 1976 (304), 18–22 (in German).—Techniques are discussed for extending the range of determinations of O with Draeger tubes (in which the O oxidises the blue - black $TiCl_3$ to the colourless $TiOCl_2$ and HCl). The coeff. of variation range from 5 to 10%, and the limit of determination is 1% (v/v) of O if 700 ml of test gas is pumped through the tube. To determine >25% (v/v) of O, two Draeger tubes are used and the sample gas is diluted 1 : 9 with air or [for >60% (v/v)] diluted 1 : 4 with N or CO_2. Oxidants (*e.g.*, Cl, Br, O_3 and NO_2) interfere. Mixtures containing compounds that ignite below 135°, such as CS_2, should not be tested, as the temp. within the tube can reach 120° owing to exothermic reaction. CHEM. ABSTR.

1J6. **Ultra-micro methods. VII. Vacuum distillation. Improvement of a previously described apparatus.** Huwyler, S. (Inst. Org. Chem., Univ. Zuerich, Switzerland). *Microchem. J.*, 1977, **22** (2), 236–237.—By dispensing with the two paper-cones and slightly tapering the distillation tube from top to bottom, use of the apparatus described in Part I (*Anal. Abstr.*, 1972, **23**, 996) is simplified so that μg amounts of material can be handled more readily. W. J. B.

1J7. **Exhaustive steam-distillation and solvent-extraction unit for pesticides and industrial chemicals.** Veith, G. D.; and Kiwus, L. M. (Environ. Prot. Agency, Res. Lab., Duluth, Minn. 55804, U.S.A.). *Bull. Environ. Contam. Toxicol.*, 1977, **17** (6), 631–636.—This apparatus (diagram given) is a modification of the steam-distillation unit used by Nielsen and Kryger (*Dan. Tidsskr. Farm.*, 1969, **43**, 39). Pesticides and industrial chemicals can be exhaustively distilled from samples of water, sediments, and tissue, with simultaneous solvent-extraction of the distillate with a small vol. of organic solvent. The extracts are suitable for direct g.l.c. Recovery of Aroclors 1016, 1242, 1248 and 1254 (0·04 μg) added to water was >98%; that of *pp'*-DDE (0·02 μg) and heptachlor epoxide (0·01 μg) was 104 and 89·9%, respectively. Recovery of Aroclor 1016 (1·7 μg g^{-1}) from river mud and of polychlorinated biphenyls (1·7 μg g^{-1}) from catfish was 78 and 84·7%, respectively, of the recovery obtained by extraction in a Soxhlet apparatus with clean-up on a Florisil column. S. M. M.

1J8. **Versatile facility for measurement of small amounts of gases in radioactive materials.** McMillan, J. W.; Weldrick, G. J.; and Taylor, B. L. (Appl. Chem. Div., AERE, Harwell, Oxon., England). *U.K. At. Energy Auth. Rep.*, AERE-R 8506, 1977. Pp. 33.—The facility includes provision for obtaining gas release thermally at temp. up to 1600° or mechanically by breaking, crushing or drilling. The equipment is shielded against radioactivity and is remotely operated. The vol. of the gas liberated is measured by using a diaphragm micromanometer and composition by m.s. Applications described include the determination of gas in coated fuel particles, pacemaker and irradiation capsules, nuclear-waste storage glass and Ni-base alloys. G. J. H.

1J9. **Automatic micro-analysers. III. For rapid micro-determination of nitrogen [in organic compounds].** Fraisse, D.; and Schmitt, N. (Serv. Central Microanal., C.N.R.S., Thiais, France). *Microchem. J.*, 1977, **22** (2), 109–130.—The completely automated system described and illustrated is based on the same principle as the Walisch apparatus (*Chem. Ber.*, 1961, **94**, 2314) and permits rapid determination of 0·5 to 100% of N in 0·5 to 2·5-mg samples. The sample is automatically released into a vertical reactor for flash combustion at 1000° to 1050°; the gases are carried in a stream of He - O (97 : 3) though a layer of CuO at 850° to 900° to ensure complete combustion, and S and halogen compounds are trapped on silvered porous Al_2O_3 pellets and silver wool at 750° to 800°. After reduction of N oxides to N, removal of O, and absorption of CO_2 and H_2O, the N in the gas stream is determined in a katharometer, with He flowing through the reference cell. Results for 25 pure organic compounds agreed closely with those obtained by other methods, and those for 25 to 30 determinations of N in atropine, phenacetin, 4-nitroaniline and thymine showed coeff. of variation ranging from 0·04 to 0·09%. The automated sample-feeder ensures repetitive determinations without the intervention of an operator. (For Part II see *Anal. Abstr.*, 1977, **32**, 1J7.) W. J. B.

1J10. **Computer automation of multi-channel Auto-Analyzers.** Ames, H. S.; and Crawford, R. W. *Am. Lab.*, 1977, **9** (2), 37–44.—Automation of the Technicon AutoAnalyzer has increased the output and accuracy of results and improved quality control in the analysis of water samples. The software (including both assembly language real-time routines and BASIC instrument programs) written for computer control of such a system in a multi-user environment is described. WRC INF.

See also Abstracts: **1B139**, Determination of NO in standard gases. **1B234**, Apparatus for isolation of water from minerals. **1C5**, Modification of apparatus for combustion of organic samples. **1C8**, Technique for weighing hygroscopic and volatile samples. **1C21**, Modified analyser for analysis of sugars. **1C90**, Distillation methods for di-isocyanates: review. **1D21**, Apparatus for collection and decomposition of organic particles. **1D30**, Ion-pair solvent extraction: review. **1D102**, Automatic system for sampling of head-space gases. **1D103**, Freeze-drying isolation of organic acids. **1E34**, Techniques for steam-distillation of essential oils. **1F47**, Steam-distillation apparatus. **1H1**, Apparatus for sampling air-borne particles. **1H2**, Sampling techniques for cotton dust. **1H3**, Preparation of dust on filters for microscopy. **1H13**, Device for sampling vinyl chloride.

Chromatography, ion exchange, electrophoresis

1J11. **Analytical gel-permeation chromatography using bead polymerised poly(acryloylprolylmorpholine).** Brough, A. W. J.; Epton, R.; McLaren, J. V.; and Marr, G. (Dept. Phys. Sci., Polytech., Wolverhampton, England). *Proc. Anal. Div. Chem. Soc.*, 1977, **14** (6), 135–137.—This material (20 to 40 μm diam.) has been evaluated for the gel-permeation separation of, *e.g.*, oligosaccharides, resin precursors and amino-acids. Elution profiles for Carbowax 200 in H_2O and in benzyl alcohol are presented to illustrate the separation efficiency attained. R. L. J.

1J12. **Simple, inexpensive method for obtaining sharp, narrow starting zones in columns packed with Sephadex gels.** Koppikar, Uday V.; Chivate, M. R.; and Koppikar, S. V. (Dept. Chem. Technol., Matunga, Univ. Bombay, India). *Anal. Biochem.*, 1977, **80** (1), 306–311.—Glass columns (50 cm × 23 mm) containing Sephadex G-200 swollen in 0·154M-NaCl were prepared to a gel height of 44·5 cm. A Millipore pre-disc filter (5 μm) of the same diam. as the column was fitted on the gel as well as an annulus with an inner-hole diam. of 20% of that of the column. Test samples of Blue dextran 2000 and cytochrome *c* were layered on to the column and eluted with 0·154M-NaCl, and 1-ml fractions were collected. R. A. H.

1J13. **Analytical studies and applications of ferroin-type chromogens immobilised by adsorption on a styrene - divinylbenzene copolymer.** Lundgren, Joel L.; and Schilt, Alfred A. (Dept. Chem., Northern Illinois Univ., DeKalb, Ill. 60115, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 974–980.—Adsorption characteristics (including the effects of pH, soln. flow rate and presence of various ions) are reported for the Fe^{II} complexes of 1,10-phenanthroline, 2,2′-bipyridyl, 2,4,6-tri-(2-pyridyl)-1,3,5-triazine and 5,6-diphenyl-3-(2-pyridyl)-1,2,4-triazine (**I**) on XAD-2 resin. Of these, **I** was the most effectively adsorbed, and columns of XAD-2 coated with **I** were successfully used to remove traces of Fe, Co, Ni and Cu from various reagent soln. and to concentrate these metals (plus Zn) by adsorption from sea-water and from soln. of $Cd(NO_3)_2$, $Cr(NO_3)_3$, ammonium acetate, chloroacetic acid and hydroxylammonium chloride for subsequent determination at sub-p.p.m. levels. R. W.

1J14. **Standardisation of test conditions for high-performance liquid chromatography columns.** Bristow, Paul A.; and Knox, John H. (ICI Ltd., Pharmaceuticals Div., Macclesfield, Ches., England). *Chromatographia*, 1977, **10** (6), 279–289.—A group of chromatographic parameters (*viz*, the reduced plate height, the reduced linear velocity of the eluent, the column flow resistance, the capacity ratio, the Knox - Parcher ratio, and the total porosity of the column) that best represents column performance for inter-laboratory comparison is recommended, standard values for these parameters are discussed, the experimental parameters for which values must be obtained are listed, and a computer program in BASIC for calculating the chromatographic parameters from the experimental ones is presented. M. J. SA.

1J15. **Siphon-type volume meter for liquid chromatography.** Weiss, Walter; Jakubik, Tibor; and Daucik, Pavol (Slovak Tech. Univ., Fac. Chem. Technol., Bratislava, Czechoslovakia). *Chem. Listy*, 1977, **71** (6), 646–648 (in Slovak).—A modified siphon trap, together with the associated electrical circuit, is described for measuring the volume of effluent from a chromatographic column. H. M. T.

1J16. **Electron-capture detector for [high-performance] liquid chromatography.** Willmott, F. W.; and Dolphin, R. J. (Mullard Research Laboratories, Redhill, Surrey, England). *Spectra 2000*, 1977, (35), 29–32.—The eluate from the column is vaporised (at 300°), and the vapour is passed directly into the detector (^{63}Ni source). Although the sensitivity is slightly poorer than that attainable with the detector in g.l.c., the system is still extremely sensitive, particularly to organochlorine pesticides. Chromatograms for pesticides in samples of surface water, domestic sewage and industrial effluents are reproduced. R. A. C. I.

1J17. **Optical detectors for liquid chromatography.** Baumann, W. (Inst. f. Phys. Chem., Univ. Mainz, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 31–38 (in German).—For photometric (filter or monochromator), polarimetric and, especially, differential refractometric detectors the sensitivities that are attained approach closely the theoretical limits. The sensitivity of fluorimetric detectors could theoretically be improved considerably, but, in view of the contamination of solvents with fluorescing substances, this would be of little value. Combinations of instruments, such as refractometer and photometer, or fluorimeter and photometer, appear to be especially useful. (8 references.) H. L. L.

1J18. **Specific detector using electrochemical technique for liquid chromatography, and its application. I. Polarographic detector with a wall-jet electrode for liquid chromatography.** Maruyama, Masao; Kakemoto, Michiko; Murakami, Kazuo; and Ishii, Toshimitsu (Fac. Sci. Eng., Chuo Univ., Tokyo, Japan). *Nippon Kagaku Kaishi*, 1977, (1), 48–53 (in Japanese).—The d.c. pulse polarographic detec-

tor described incorporated a wall-jet disc electrode (planar; vitreous carbon) as working electrode, a tubular counter-electrode and a s.c.e. as reference. The detector was evaluated by use of nitrobenzene (in electro-reduction) and aniline (in electro-oxidation) as test substances. The linear dynamic range between limiting diffusion current and concn. was $\simeq 10^3$, and the minimum detectable amount was 1·2 ng for nitrobenzene and 1·9 ng for aniline. The limit of detection with this detector was similar to that with a u.v. detector. Applications of the polarographic detector include accurate determination of electro-active compounds in multi-component samples. CHEM. ABSTR.

1J19. **Mercury-based electrochemical detector for liquid chromatography for detection of glutathione and other sulphur-containing compounds.** Rabenstein, Dallas L.; and Saetre, Rolf (Dept. Chem., Univ. Alberta, Edmonton, Canada). *Anal. Chem.*, 1977, **49** (7), 1036–1039.—The detector comprises a mercury-pool working electrode (0·8 mm diam.), and has a channel through which the column effluent passes, a channel perpendicular to and in contact with the effluent channel for the electrode, and a well for a reference electrode. Electrical contact between the reference and working electrode is accomplished by another channel. It has a potential range from $\simeq +0{\cdot}2$ V to $\simeq -1{\cdot}2$ V *vs.* the s.c.e. depending on pH. As well as glutathione, the detector has been used in the determination of, *e.g.*, S-containing amino-acids, inorganic S compounds and thiourea. G. P. C.

1J20. **Lanthanum tellurate, a new inorganic ion exchanger.** Gonzalez, Conceicao G.; and Carvalho, R. A. Guedes de (Centro de Eng. Quim., Fac. Eng., Porto, Portugal). *J. Chromatogr.*, 1977, **136** (1), 176–177.—The $La_2(TeO_4)_3$ was prepared by adjusting a soln. of $LaCl_3$ and $Te(OH)_6$ (La-to-Te ratio 2:3) to pH 4·5 with aq. NH_3, filtering and washing, and drying the ppt. *in vacuo* at 50°. The variation with pH in the exchange capacity of $La_2(TeO_4)_3$ for Na^+ was determined. M. J. SL.

1J21. **Comparative study of new carboxylic cation exchangers.** Makarova, S. B.; Bezuevskaya, S. I.; Sedel'nikova, G. V.; Ivanovskii, M. D.; and Egorov, E. V. (All-Union Sci.-Res. Inst. Reagents and High-purity Chemicals, Moscow, USSR). *Zh. Anal. Khim.*, 1977, **32** (5), 892–896 (in Russian).—Two cation exchangers (KRK and KRDK) based on styrene-divinylbenzene copolymers and containing 4-carboxyphenyl (KRK) or 3,4-dicarboxyphenyl (KRDK) residues have been prepared and examined as sorbents for Ag^I, Co^{II}, Cu^{II}, Ni^{II} and Fe^{III}. The exchange capacities (m-equiv. g^{-1}) were 4 to 5 for KRK and 6 to 7 for KRDK, the former having a pK value of 6·4 and the latter pK values of 3·2 and 6·2. The KRDK exchanger (H^+ form) can be used for the selective separation of mg amounts of Cu^{II} from Ag^I; when the test soln. (at pH 4 to 5) is set aside with the resin for at least 30 min, the Cu is adsorbed and the Ag is not. M. P.

1J22. **Synthesis and properties of a cellulose ion exchanger with a tiron residue as functional group.** Burba, P.; Griesbach, M.; and Lieser, K. H. (Fachber. Anorg. Chem. u. Kernchem. Tech. Hochsch., Darmstadt, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **284** (4), 257–261 (in German).—Procedures are described for preparing the exchanger and for determining its capacity. Distribution coeff. are reported for Cu^{2+}, Hg^{2+}, Fe^{3+} and the alkaline-earth-metal ions in the pH range 0 to 3, and results are discussed in terms of the bonding mechanism. C. K. L.

1J23. **A vertical-slab electrophoresis apparatus.** Sauaia, H.; and Laicine, E. M. (Dept. Morfol., Fac. Med. Ribeirao Preto, Univ., Sao Paulo, Brazil). *Anal. Biochem.*, 1977, **80** (1), 125–132.—A new design for such an apparatus, constructed in glass or plastic, is described and shown in diagrams. The use of the device is exemplified by the separation of extracts of dipterean salivary glands by discontinuous electrophoresis on SDS-polyacrylamide gels. R. A. H.

1J24. **Achievement of a rectilinear gradient of range one pH unit in isoelectric focusing.** Viau, M.; Constants, J.; and Bouissou, C. (C.N.R.S., Centre d'Hemotypol., Hopital Purpan, Toulouse, France). *Sci. Tools*, 1977, **24** (2), 25–26.—The cathodic drift of ampholytes is controlled in a migration by using a current of 30 mA (at 12°) until the max. voltage of 1000 V has been reached and then allowing it slowly to decrease to settle at 20 mA. A rectilinear gradient of pH 4 to 5 over the whole surface of the gel is thereby obtained after 4·5 h from Ampholine of pH range 4 to 6. The method has been used to study the polymorphism of α_1-antitrypsin. H. K. M.

1J25. **Sample applicators for thin-layer chromatography.** Chobanov, D. G. (Inst. Org. Chem., Acad. Sci., Sofia, Bulgaria). *Lab. Pract.*, 1977, **26** (6), 481.—The applicators are constructed by gluing one strip of glass, having a sharp uniform edge, on either side of the end of a square-sectional glass rod that has been cut to a wedge shape. The edges of the two strips (which are inclined to one another at an angle of 25°) form a gap 0·2 mm wide; sample soln. spreads along these edges by capillary action. Repeated application does not damage the adsorbent layer. Constructional diagrams are given for several variants, including multiple-sample applicators. S. M. M.

1J26. **Preparation of plates with a permanent adsorbent layer and their use in the analytical thin-layer chromatography of lipids.** Tysdendambaev, V. D.; Zhukov, A. V.; and Vereshchagin, A. G. (K. A. Timiryazev Inst. Physiol., Moscow, USSR). *Fiziol. Rast. (Moscow)*, 1977, **24** (2), 437–443 (in Russian).—Window glass was ground (to 195 mesh) in a ball mill; it was then mixed with silica gel in the ratio 3:1 (w/w). A toluene suspension of the mixture was layered on a glass plate, which was then heated (initially at 100° to 150°, and then at 675° for 20 min). The plate performed as well as conventional adsorbent layers for the separation of lipids and could be used again after treating it with H_2SO_4 - HNO_3 (9:1) for 30 min at 100° to 120°, then washing with water and activating at 110° for 30 min. CHEM. ABSTR.

1J27. **Spray technique for applying stripmix (cellulose acetate) to thin-layer chromatography plates for tritiated organic compounds.** Forti, Giorgio Cantelli; Trieff, Norman M.; Ramanujam, V. M. Sadagopa; and Kilian, D. Jack (Dept. Preventive Med. and Community Health, Univ. Med. Branch, Galveston, Tex. 77550, U.S.A.). *Anal. Biochem.*, 1977, **80** (1), 319–321.—A modification of the 'rolling' technique of Redgwell *et al.* (*J. Chromatogr.*, 1974, **88**, 25) is described. Tritiated benzo[*a*]pyrene and its metabolites (from the aryl hydrocarbon monooxygenase assay) were separated

by t.l.c. on starch-bound silica gel with benzene - hexane (1 : 15) as developing solvent. Cellulose acetate - acetone (7 : 3) was sprayed on to the plates, and, after drying the chromatogram for 15 min, the spots revealed were excised and placed in phials containing scintillator soln. for counting. Highly reproducible results were obtained. R. A. H.

1J28. **Control apparatus for automatic, spectrophotometric, direct evaluation of thin-layer chromatograms.** Pohl, Uwe; Schweden, Werner; Lessnig, Werner; and Metz, Guenter (ZF-WHL-Ue-Analytik, Bayer A.-G., Krefeld-Uerdingen, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **285** (2), 111–120 (in German).—An instrument is described for the automated measurement of up to 8 traces each containing up to 10 components on a t.l.c. plate (200 mm × 200 mm) within 11 min. The sensing head is driven by a stepping motor allowing precise positioning. The apparatus, which has a digital output to allow computer processing of data, may also be used manually. A number of arithmetic functions are considered for calibration; the Kubelka - Munk function is preferred, giving a rectilinear calibration over a 100-fold concn. range. The precision and accuracy of the equipment were demonstrated on a test mixture of dyes. A coeff. of variation of $\simeq 2\%$ was obtained when the apparatus was used in conjunction with a band-forming loading apparatus. This was twice as good as that obtained from manual positioning on spots. D. B. A.

1J29. **Correct measurement and interpretation of evaluation factors in chromatography: the real plate number, the separation number, the dosage quality. I. Gas chromatography.** Kaiser, Rudolf E. (Inst. for Chromatogr., Bad Duerkheim, W. Germany). *Chromatographia*, 1977, **10** (6), 323–338.—By the proposed method, 'real' plate numbers can be calculated that are independent of the nature of the test substance, of the method of injection and of the column connections. (A German version of this paper appeared in *Ibid.*, 1976, **9**, 337.) M. J. Sa.

1J30. **Mass-spectrometric quantitation in gas chromatography, and other gas-chromatography advances.** Millard, Brian J. (Sch. Pharm., Univ. London, Brunswick Sq., London, W.C.1, England). *Methodol. Dev. Biochem.*, 1976, **5**, 1–7.—In this review, the advantages and limitations of open-tubular columns in comparison with packed columns are discussed, and detection by m.s. is compared with electron-capture, flame-photometric, coulometric and thermionic detection. The choice of internal standard when m.s. is used for detection is also considered. (13 references.) B. H.

1J31. **An inexpensive automated recycle radio-gas chromatograph.** Lambrecht, R. M.; Withnell, R.; and Wolf, A. P. (Chem. Dept., Brookhaven Natl. Lab., Upton, N.Y. 11973, U.S.A.). *Chem. Instrum.*, 1976, **7** (3), 157–170.—In the instrument described, the 2 chromatographic columns are connected to an 8-port, 2-position microvolume switching valve. The multi-timer circuit (diagram given), which controls this valve, comprises 20 integrated circuits. The instrument is designed for routine analytical separations (of, *e.g.*, simple hydrocarbons labelled with ^{11}C), and the operator need only inject the sample and start the multi-timer. When radioactive compounds are to be assayed, the apparatus is used with a flow proportional counter, and, preferably, an absolute flowmeter: the observed counts are easily corrected for counting efficiency perturbations. The detection efficiencies for ^{11}C and ^{35}S are listed for various Mylar, Hyvar, aluminium, titanium and nickel windows in the flow proportional counter. L. B.

1J32. **Connection of glass capillary columns to a Pye series 104 chromatograph.** McGill, Alister S.; Parsons, Eva; and Smith, Alistair (Torry Res. Sta., Aberdeen, Scotland). *Chem. Ind.*, 1977, (11), 456–457.—Best results are obtained when the column inlet is connected to the injection block by means of a length of silanised soda-glass tubing (70 mm × 0·3 mm i.d.) joined to the column by heat-shrunk PTFE tubing and so positioned that its upper portion is heated by the injection-port heater. A similar tube (145 mm long) encased in stainless-steel tubing is used to connect the column outlet to the flame ionisation detector; it is so positioned that its tip is above the H inlet, but below the flame. Graphite ferrules are used to effect connection at the inlet and outlet ends. B. H.

1J33. **Nanogram techniques. I. Injection without losses on capillary gas - liquid chromatographic columns.** Klimes, I.; Stuenzi, W.; and Lamparsky, D. (Givaudan Forschungsgesellscahft A.-G., Duebendorf, Switzerland). *J. Chromatogr.*, 1977, **136** (1), 13–21 (in German).—In order to inject ng amounts of sample on to the column without losses incurred by the use of by-pass splitters, an adapter to take micro-syringes is fitted to the injection port. It consists of a short length of capillary tubing with a constriction in the middle held in a glass insert in the port so as to fit over the end of the capillary column. Also a 1-μl Hamilton syringe is adapted by removing about 10 mm from the end of the needle by an oblique cut, and the projecting 10 mm of the wire plunger is reduced in diam. by rubbing down with emery paper. By withdrawing the plunger, a sample of liquid or head-space vapour, *e.g.*, from a single bloom, can be drawn into the narrow annular space between the end of the plunger and the needle and injected into the adapted port. Substances with b.p. $>220°$ are introduced in short lengths of capillary tubing used as condensing traps for vaporisation in the injection port. R. F. S.

1J34. **Simple micro-reactor for subtractive gas chromatography.** McKeag, R. G.; and Hougen, F. W. (Dept. Plant Sci., Univ. Manitoba, Winnipeg, Canada). *Anal. Chem.*, 1977, **49** (7), 1078–1079.—A micro-reactor is described that can fit on to the injection port of a conventional gas chromatograph; it comprises a simple tube (filled with an adsorbent and plugged with glass wool) connected to the column by a standard Swagelok coupling. The tube can be operated at a temp. different to that of the column, and its use is exemplified by the adsorption of alcohols on H_3BO_3, of aldehydes on *o*-dianisidine or benzidine and of ketones on benzidine. R. W.

1J35. **Problems in choosing a stationary phase for gas-chromatographic separation. II.** Lekova, K. *Khim. Ind.* (*Sofia*), 1977, **49** (6), 254–260 (in Bulgarian).—This review deals with the different types of compound proposed as stationary phases for g.l.c. in terms of their Rohrschneider and Reynolds constants. Standard substances suggested for the various required types of phase are tabulated, and chromatographic equivalents of these standards are listed (often under trade names). Numerical analysis (by computer) of retention data for specific

substances and specific stationary phases is suggested as a promising way of selecting a stationary phase for a particular separation. (48 references.)
C. D. K.

1J36. **Characteristics of poly(trimethylene oxide) as a liquid phase for gas chromatography.** Tsuge, S.; Fujimoto, T.; Miyahara, N.; Kobayashi, T.; and Takeuchi, T. (Dept. Synth. Chem., Nagoya Univ., Japan). *J. Chromatogr. Sci.*, 1977, **15** (2), 73–74.—This liquid phase is prepared by ring-opening polymerisation of trimethylene oxide (oxetane) and has the structure $HO(-CH_2CH_2CH_2O-)_nH$. Its polarity is intermediate between that of polyoxypropylene and polyoxyethylene glycols, but it is thermally more stable (usable up to 270°). The advantages of this liquid phase were demonstrated by the separation of a mixture of C_1 to C_{10} alcohols. M. J. Sa.

1J37. **Check lists on errors and their causes in gas chromatography with different types of detector.** Franke, Wolfgang (Deutsche L'Air Liquide Edelgas G.m.b.H., Duesseldorf, W. Germany). *GIT Fachz. Lab.*, 1977, **21** (6), 488–489 (in German).—Charts are presented that relate defects observed in chromatograms to conditions applied in the analysis, *e.g.*, flow of carrier gas and column temp., for thermal-conductivity, flame ionisation and high-frequency discharge detectors. W. C. D.

1J38. **Reaction mechanism in an ionisation detector with tunable selectivity for carbon, nitrogen and phosphorus.** Kolb, B.; Aver, M.; and Pospisil, P. (Bodenseewerk Perkin-Elmer & Co. G.m.b.H., Ueberlingen, W. Germany). *J. Chromatogr. Sci.*, 1977, **15** (2), 53–63.—The commercially available N - P detector, based on a rubidium silicate bead to achieve selectivity, is modified so that it can be used in the conventional flame ionisation mode. The change-over between the various modes is claimed to be simple and quick. Possible mechanisms for the N - P detection are discussed. Applications of the detector to the analysis of N-containing organic compounds, drugs in body fluids and essential oils are given. M. J. Sa.

1J39. **Instrumental effects on limits of detection in gas-phase fluorescence detection of gas-chromatographic effluents.** Cooney, R. P.; and Winefordner, J. D. (Dept. Chem., Univ. Florida, Gainesville, Fla. 32611, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1057–1060.—The systems examined incorporate variously a 150-W Eimac Xe-arc lamp (pulsed and continuous), a 200-W Hg - Xe arc and a pulsed N laser, with the quartz flow-through detector cell being positioned inside the chromatograph oven in some instances. Limits of detection with the different systems are reported for anthracene, pyrene, chrysene and phenanthrene. Improved performance can be achieved by use of sources with intense u.v. output and by use of excitation monochromators with lower stray-light levels. R. W.

1J40. **Application of a micro carbon-rod atomiser as bromine-specific gas-chromatographic detector.** Gutsche, B.; Ruediger, K.; and Herrmann, R. (Abt. Med. Phys., Univ. Giessen, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **285** (2), 103–106.—The exit gas from a g.l.c. column is passed into the base of a modified graphite cell mounted between the electrodes of a Varian carbon-rod atomiser (model 63). The cell contains indium and is heated to 430° by the slightly modified control unit. An Ar - H flame burns continuously around the cell, a small silica chimney producing an even flame. Bromine compounds, *e.g.*, bromoethane (**I**) or 1,2-dibromoethylene (**II**), entering the cell result in the formation of InBr. The InBr is excited in the flame to give a spectrum with max. emission at 372·7 nm, which is measured photometrically. The detection limits for **I** and **II** are 5 and 36 ng of Br, respectively, with a precision of ±4·3%. Calibration is rectilinear from 0·01 to 2 μg of Br for **I** and 0·035 to 4 μg of Br for **II**. D. B. A.

1J41. **Application of the microwave emission spectrometric system as a silicon-selective detector.** Bostick, D. T.; and Talmi, Yair (Anal. Chem. Div., Oak Ridge Natl. Lab., Tenn. 37830, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (5), 164–167.—The microwave emission spectrometric - g.l.c. system used was similar to that described by McCormack *et al.* (*Anal. Chem.*, 1965, **37**, 1470). Three types of discharge have been tested and optimal performance was achieved with Ar at 3 to 5 Torr with use of the Si line at 251·6 nm. When a 6-ft.-long glass column packed with 4% of OV-101 on Chromosorb G HP (80 to 100 mesh) was used, at 250° with Ar as carrier gas (80 ml min^{-1}), the detector response to tributylsilane was rectilinear over the range 0·5 to 150 ng of Si with coeff. of variation of 2 to 3%. The response to the trimethylsilyl ester of octanoic acid was rectilinear for 1 to 60 ng of Si; the selectivity ratio of such esters to linear alkanes was ≃100. The g.l.c. system could be operated in the double detection mode using a flame ionisation detector with the microwave emission spectrometer. M. J. Sl.

1J42. **Nanogram techniques. II. Micro-scale preparative chromatography.** Klimes, I.; Stuenzi, W.; and Lamparsky, D. (Givaudan Forschungsgesellschaft A.-G., Duebendorf, Switzerland). *J. Chromatgr.*, 1977, **136** (1), 23–36 (in German).—Traps consisting of capillary m.p. tubes containing small lengths of cotton-wool, and cooled in a solid CO_2 - liquid N bath, are fitted at the ends with short lengths of PTFE tubing, so as to make gas-tight fits when inserted into the conical orifice of the flame ionisation detector outlet port. In this way, the preparative isolation of μg to mg amounts of substances separated on packed or capillary analytical columns is possible. (For Part I see *Anal. Abstr.*, 1978, **34**, 1J33.) R. F. S.

1J43. **Coupling of a capillary gas chromatograph to a mass spectrometer having provision for chemical ionisation, and the recording of the chromatogram with a spectrum integrator.** Matthiesen, U.; Kump, B.; and Staib, W. (Inst. Physiol. Chemie, Univ. Duesseldorf, W. Germany). *Chromatographia*, 1977, **10** (6), 303–308 (in German).—The construction of such a coupling device is described; its use permits both electron-impact and chemical-ionisation spectra to be recorded without major alteration to the apparatus. The ion current can be integrated over each mass scan and the resulting output fed to the recorder. M. J. Sa.

1J44. **Trap - splitter unit for coupling preparative gas-chromatographic columns to analytical capillary columns.** Batiz, Heriberto; and Rosado, Eduardo (Rum Pilot Plant, Agric. Exp. Stn., Rio Piedras, Puerto Rico). *J. Agric. Univ. P.R.*, 1977, **61** (1), 108–112.—Trace components separated by preparative g.c. can subsequently be detected by analytical g.c. on capillary columns by using a trap - splitter unit having a trap made from a stainless-steel U-tube (5 mm i.d.) that allows the collection of >10-μl

fractions without noticeable interruption of the carrier-gas flow. Liquid N or solid CO_2 is used as coolant. For analysis, the collected fraction is evaporated by means of a flash heater; a 1:63 splitter ratio is used. The apparatus has been used to analyse a mixture of alcohols and acetates.

CHEM. ABSTR.

1J45. **Concentration in gas chromatographic - mass spectrometric analysis by using a scanning separator.** Grishin, V. D.; Galushkin, B. A.; and Tal'roze, V. L. (Inst. Chem. Phys., Acad. Sci. USSR, Moscow). *Zh. Anal. Khim.*, 1977, **32** (6), 1061–1065 (in Russian).—The separator described for concentrating components of g.c. effluents before they enter the detector or a mass spectrometer consists of a U-tube ($\simeq$10 to 100 cm × 3 to 10 mm) that is alternately cooled and heated by a rotating disc (moving slowly in the reverse direction to the carrier-gas flow) having thermally insulated heater and freezer segments. The compounds are individually frozen after leaving the g.c. column at a carrier-gas flow rate of $\simeq$100 ml min^{-1}, and are then sequentially passed to the detection system with a flow rate of $\simeq$0·1 ml min^{-1}. No losses are caused, and the enrichment factor is 100 to 1000 (depending on the ratio of the flow rates).

M. P.

1J46. **Real-time analysis of gas chromatograms with a micro-computer system.** Bobba, G. Mario; and Donaghey, Lee F. (Mater. and Mol. Res. Div., Univ. California, Berkeley, Calif. 94720, U.S.A.). *J. Chromatogr. Sci.*, 1977, **15** (2), 47–52.—A program is developed by which the start of peaks, the point of max. and the end of peaks can be detected by statistical means. The micro-computer system uses an Intel 8008 microprocessor in a bin-oriented structure. The accuracy of chromatographic peak integration is studied with propane - He mixtures on a gas chromatograph with a thermal-conductivity detector. The results of the on-line system are compared with those of an off-line system. Slightly lower results are obtained by the former procedure, which is considered to be due to differences in the mathematical sub-routines and the detection of the integral end-point.

M. J. SA.

See also Abstracts: **1B6**, Silica support for chromatography of metal ions. **1B7**, Treated paper for chromatography of metal ions. **1B9**, System for extraction - electrophoresis of metals. **1B115**, Apparatus for g.c. with circulation of carrier gas. **1B145**, Formation of derivatives for g.l.c. of organic acids. **1B223**, Electrophoresis of Pd and Pt on impregnated paper. **1C1**, High-efficiency columns for gel-permeation chromatography. **1C12**, Use of ion exchange in determination of S and Cl. **1C21**, Automatic ion-exchange and gel-permeation chromatography of sugars. **1C30**, Stationary phases for g.l.c. of various organic compounds. **1C31**, Impregnated paper for chromatography of amines. **1C84**, Silica packings for h.p.l.c. of organic compounds. **1C90**, Chromatography of di-isocyanates: review. **1D2**, Chromatographic analysis of biological samples: review. **1D3**, Technique for isotachophoresis. **1D7**, Computerised g.c. of cigarette smoke. **1D30**, Chromatography in biochemical analysis: review. **1D38**, Evaluation of detector for g.l.c. of drugs. **1D102**, Automatic system for sampling of head-space gases. **1E1**, Response of selective g.c. detector to drugs. **1F3**, Determination of trace contaminants in food by g.l.c.: review. **1H11**, Adsorbent for g.c. of organic compounds. **1H32**, Ion-exchange isolation of U from water. **1H43**, Materials for collection of pollutants from water. **1J86**, Fluorescence detectors for liquid chromatography. **1J90**, Raman detector for liquid chromatography. **1J96**, M.s. of g.l.c. effluents. **1J104**, Electrolytic chromatography: review.

Spectroscopy

Including spectra by absorption, emission, reflection or magnetic resonance; optical properties; mass spectra

1J47. **Recommended reference materials for realisation of physico-chemical properties. Recommendations (1976). Section: absorbance and wavelength.** International Union of Pure and Applied Chemistry, Physical Chemistry Division, Commission on Physicochemical Measurements and Standards, Subcommittee on Calibration and Test Materials. *Pure Appl. Chem.*, 1977, **49** (5), 661–674.—This report has sections on reference materials for (*i*) absorbance/transmittance measurements, and (*ii*) wavelength/wavenumber measurements, for use in the u.v., visible and i.r. regions. Sources of supply and/or methods of preparation of the materials are included, together with pertinent physico-chemical data.

R. L. J.

1J48. **Investigation of thin layers with the scanning electron microscope [SEM].** Holm, R.; and Storp, S. (Ing.-Ber. Angew. Phys., Bayer A.-G., Leverkusen, W. Germany). *Mikrochem. Acta*, 1977, Suppl. VII, 171–184 (in German).—Surface layers of 10 to 100 nm can be examined by SEM, while thinner layers require changes in the primary-beam voltage or the use of taper sections. For the analysis of monolayers, a combination of SEM with an Auger spectrometer is recommended.

J. W. P.

1J49. **Colour techniques in scanning electron microscopy and micro-analysis.** Hoerl, E. M. (Studiengesellschaft fuer Atomenergie G.m.b.H., Seibersdorf, Austria). *Mikrochim. Acta*, 1977, Suppl. VII, 197–207 (in German).—A review is presented in which the techniques at present in use are discussed. Advantages over the 'black-and-white' techniques are outlined. (27 references.)

J. W. P.

1J50. **Automated chemically specific metallography: hardware, software and applications.** Malissa, H.; Grasserbauer, M.; Terenyi, W. M.; Hoke, E.; and Reisenhofer, K. (Inst. Anal. Chem., Tech. Univ., Vienna, Austria). *Mikrochim. Acta*, 1977, Suppl. VII, 501–521 (in German).—Automated equipment for linear analysis by micro-probe is described and its use is exemplified by the examination of four samples of a Cr - Ni - Mo steel containing 0·5 to 1·9% of B.

J. W. P.

1J51. **Is optical emission spectrometry being tailored to the requirements and budgets of a large variety of users?** Boumans, P. W. J. M. (Philips Research Lab., Eindhoven, Netherlands). *Proc. Anal. Div. Chem. Soc.*, 1977, **14** (6), 143–151.—The review (with 102 references) summarised here was presented as the plenary lecture at a joint meeting of the North East Region and the Atomic Spectroscopy Group of the Analytical Division of the Chemical Society, which was held at Middlesbrough, England, in October 1976.

1J52. **Non-linear least-squares method of determining line intensities and half-widths.** Chang, Y. S.; and Shaw, John H. (Dept. Phys., Ohio State Univ., Columbus, Ohio 43210, U.S.A.). *Appl. Spectrosc.*, 1977, **31** (3), 213–220.—A method of calculation (suitable for use with a computer) is described that requires only observation of the spectrum and knowledge of the amount of absorbing gas present (in atm cm at s.t.p.), the spectral line shapes and the response function of the instrument, and that can be applied to single or overlapping lines. A number of parameters can be determined, including the line intensity and half-width, the position of the line centre, the spectral width of the response function, and the spectrometer response both in the absence of any absorber and when absorption is complete at all wavelengths.
M. A. P.

1J53. **Mathematical evaluation of X-ray valency band spectra.** Drack, Helmut; and Grasserbauer, Manfred (Inst. Anal. Chem. and Microchem., Tech. Univ., Vienna, Austria). *Mikrochim. Acta*, 1977, Suppl. VII, 289–302 (in German).—Identification of compounds can be made easier by enhancement of the fine structure by smoothing, differentiation and unfolding of spectra by a mathematical treatment. The method is exemplified by analysis of the spectra for Cu, Cu_2O and CuO.
J. W. P.

1J54. **Two-plate multiplier for the ultra-soft region of X-rays.** Schnell, W.; and Wiech, G. (Sekt. Phys., Univ. Muenchen, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 323–344 (in German).—The construction of a detector for X-rays of wavelength >20 Å is described. The electric field in the multiplier is generated by a high-resistance evaporated film for which carbon, silicon or germanium can be used. The resistance layer is covered with a thin film of alumina, which yields the secondary electrons, and an evaporated film of gold serves as the photo-cathode. Ways of optimising the instrumental performance are discussed.

1J55. **Preparation of solid standard samples for X-ray fluorescence analysis.** Revin, Yu. V.; Ryabinin, V. B.; and Larin, N. V. (Inst. Chem., Acad. Sci. USSR, Gor'kii). *Zh. Anal. Khim.*, 1977, **32** (6), 1237–1240 (in Russian).—Homogeneous standard samples of zinc, germanium or copper, with the addition of 0·1 to 10% of Al, Sb or Ce_2O_3, were fused in silica crucibles to obtain compact raw beads (5 to 7 g). Each bead was fused while suspended in a h.f. electromagnetic field (*cf. Anal. Abstr.*, 1976, **30**, 5B152) in an atmosphere of He for ≃1 min, and the melt was cast in a silica dish to form a disc (diam. 1 cm; height 6 mm).
M. P.

1J56. **Improved method for calculating the intensity of fluorescent X-radiation of wavelength up to 3 Å.** Vener, B.; Kleinstueck, Karl H.; and Moras, K. (Dresden Tech. Univ., E. Germany). *Zavod. Lab.*, 1977, **43** (6), 675–680 (in Russian).—The proposed method, applicable to samples containing at least three elements, takes into consideration primary, secondary and inter-element tertiary radiation. It has been used in determining Cr (in Ni - Fe - Cr alloys) from the $K\alpha$ radiation excited by emission from a molybdenum-target tube. The advantages and limitations of the method are discussed.
A. S. W.

1J57. **X-ray fluorescence analysis without reference samples.** Ebel, Horst (Inst. Tech. Phys., Tech. Univ., Vienna, Austria). *Mikrochim. Acta*, 1977, Suppl. VII, 545–553 (in German).—The effect of the amplitude factor of the X-ray-tube spectrum on count rates can be eliminated by comparison of count rates measured under two different geometries. The procedure is applied to Cu coatings on Ag and vice versa.
J. W. P.

1J58. **Matrix corrections in trace-element analysis by X-ray fluorescence: extension of the Compton-scattering technique to long wavelengths.** Nesbitt, R. W.; Mastins, H.; Stolz, G. W.; and Bruce, D. R. (Dept. Geol. and Miner., Univ. Adelaide, S. Australia). *Chem. Geol.*, 1976, **18** (3), 203–213.—A modification of the Compton-scattering method for estimating mass-absorption coeff. is described for the range 1·79 to 3·03 Å. The coeff. have been calculated for the range 0·492 to 3·03Å and are shown to be rectilinearly related to the Compton-peak intensity for wavelengths shorter than the Fe absorption edge at 1·74 Å. At longer wavelengths, the coeff. can be estimated from measurement of the intensity of the Fe $K\alpha$ peak and the Compton scatter, the latter being measured by using a thick sample of MgO or quartz. This technique enables matrix corrections to be performed for Co, Mn, Cr, V, Ti, Si (K spectra) and Ba (L spectra). Values are given for Ba and Cr for 11 standard rocks.
M. A. P.

1J59. **Statistical errors and resolution limits in energy-dispersive X-ray analysis.** Ryder, P. L. (Fachber. Phys., Univ. Bremen, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 231–242 (in German).—A mathematical expression is derived for the quantitative evaluation of experimental errors due to background and to line position and width; an assessment of the attainable accuracy of measurements is thus provided.
J. W. P.

1J60. **Program for calculating relative intensities of lines in X-ray-fluorescence analysis.** Lavrent'ev, Yu. G.; and Kuznetsova, A. I. *Tr. Inst. Geol. Geofiz. Akad. Nauk SSSR, Sib. Otd.*, 1977, (315), 60–70 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G48.

1J61. **Bremsstrahlung background from massive samples in electron-probe micro-analysis.** Boecker, J.; and Hehenkamp, T. (Inst. Metallphys., Univ. Goettingen, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 209–230 (in German).—A method was developed for the calculation of background correction by measurements with pure metals and binary alloys. An accuracy of 99% was attained.
J. W. P.

1J62. **Non-dispersive X-ray diffractometry with semiconductor detectors.** Mantler, M. (Inst. Tech. Phys., Tech. Univ., Vienna, Austria). *Mikrochim. Acta*, 1977, Suppl. VII, 555–566 (in German).—It is shown that width, asymmetry and shift of a non-dispersive diffraction line increase with increasing energy, beam divergence and specimen thickness and with decreasing Bragg angle.
J. W. P.

1J63. **High-purity water for atomic-absorption analyses.** Sampson, R. *Am. Lab.*, 1977, **9** (3), 81–84.—The impurities, especially trace elements, present in distilled and de-ionised water are discussed and a water-purification system is described.
WRC Inf.

1J64. **Ultra-violet photo-electric spectra from some microwave lamps suitable for calibrating low- and medium-resolution spectroscopic instruments between 180 nm and 500 nm.** Freeman, G. H. C.; and

Freeman, I. (Div. Quantum Metrol., Natl. Phys. Lab., Teddington, Middx., England). *Natl. Phys. Lab. (U.K.), Rep.*, QU 35, 1977. Pp. 27.—Small, commercially available lamps intended for a.a.s. and emitting a few suitably spaced spectral lines have been considered. All the observed lines of reasonable intensity are listed for He, Ne, Ar, Kr, Xe, Zn, Cd, Hg, I, S, Cu, Ge, H, Fe, Th and Se; impurity lines are included. The spectrograms of all the cited elements except Se are reproduced to indicate the usefulness of the spectra. R. L. J.

1J65. **Refractory analysis: prevention of sample contamination by using pyrolytic-graphite-coated furnace sampling tubes.** Manning, D. C.; Fernandez, F. J.; and Peterson, G. E. (Perkin-Elmer Corp., Norwalk, Conn. 06856, U.S.A.). *Ind. Res.*, 1977, **19** (2), 82–85.—A procedure for the coating of atomic-absorption tubes by pyrolysis of methane is described. This enhances to different degrees the signals due to B, Cu, Mn, Mo, Ti and V (but not Al) and increases the useful lifetime of the tube, especially at high temp. (up to 3000°) or with samples having high concn. of acid. For Pb, Cd and Au, there is improved sensitivity in aq. soln. but poorer sensitivity in organic soln. For the determination of Pb in blood, the precision of analysis by using pyrolytic and standard tubes is essentially the same. Sensitivity, especially to B, is further improved by rapid heating, and signals due to B are enhanced by the addition of Ca, Mg, Sr or La. The determination of B in orchard leaves and fertilisers is described. L. B.

1J66. **Determination of concentration of atoms in a plasma.** Baranova, I. D.; and Tskhai, N. S. *Zh. Prikl. Spektrosk.*, 1977, **26** (3), 413–416 (in Russian).—The method described involves measuring the integral absorption of the atoms by trans-illuminating the radiation source with reflected radiation from the same source. A scheme of the optical system of the spectrograph is given: the spectra as photographed with and without trans-illumination are compared. The method was demonstrated by determining Zn, in the plasma of a 5-A a.c. arc between Cu-alloy electrodes, from the integral absorption at 307·6 nm. The concn. of Zn in the plasma was plotted as a function of Zn concn. in the electrode. CHEM. ABSTR.

1J67. **Improved accuracy in spectral analysis of powders blown into an arc discharge.** Raikhbaum, Ya. D.; Popyalkovskaya, L. K.; and Somina, L. A. (Gos. Univ. im. Zhdanova, Irkutsk, USSR). *Izv. Vyssh. Uchebn. Zaved., Fiz.*, 1977, **20** (1), 78–84 (in Russian).—It is shown that systematic errors in the spectrography of powder samples blown into an arc discharge could be diminished by a factor of 1·5 to 2 by taking account of the shape of the intensity distribution of the analytical line along the discharge axis. The arc was sub-divided into two parts and the intensities (I_1 and I_2) in these parts were measured by recording two spectrograms simultaneously. The asymmetry factor (S) and the corresponding analytical intensity were calculated as $(I_1 - I_2)(I_1 I_2)^{0.5}$ and $(I_1 + I_2)(1 - S)$, respectively. The method was applied in determining traces of Pb, Sn and Zn in minerals, soils and plants. CHEM. ABSTR.

1J68. **Spectral characteristics of a discharge in a narrow channel as excitation source for analysis of solutions.** Krasil'shchik, V. Z.; Raginskaya, L. K.; and Manova, T. G. *Zh. Prikl. Spektrosk.*, 1977, **26** (3), 408–412 (in Russian).—A study is presented of the use of an a.c. discharge in a narrow channel as a source for spectrographic analysis of dil. soln., with particular reference to the effects of potential (1500 to 3000 V), geometrical parameters, and the nature of the test liquid. The spectral intensities (highest in the central zone of the discharge) for most trace elements depended on the soln. matrix, decreasing in the order HCl, HNO_3, H_2SO_4, H_3PO_4; the highest intensities were observed with 5% HCl. Organic compounds and NH_3 increased the intensities. The sensitivities (p.p.m.) for determining transition metals, Group IIIA metals, Pb, alkali metals and alkaline-earth metals were ≃0·1, 0·1, 10, 1, 0·1 and 0·01 in media of HCl, $MgCl_2$, $AlCl_3$, $NiCl_2$, citric acid and NH_3, respectively. CHEM. ABSTR.

1J69. **Effects of potassium chloride on the analytical characteristics of the capillary arc excitation source.** Kubota, Masaaki; Ishida, Ryohei; and Fujishiro, Yoshimasa (Natl. Chem. Lab. Ind., Shibuya-ku, Tokyo, Japan). *Bunko Kenkyu*, 1977, **26** (2), 63–69.—Diagrams are given of such a source incorporated into a complete analytical system. A soln. of metal ions is aspirated, after desolvation, into the source with a stream of Ar (1·15 l min^{-1}). When the soln. is 0·1M in KCl, the background emission is reduced and the signal enhanced. For Al, Fe, Ga, Ni and Zn, this results in a 10-fold increase in the signal-to-noise ratio. Limits of detection are quoted for 19 elements; all except those of W and Zr are improved by use of KCl soln. Calibration graphs are rectilinear and coeff. of variation are <2% for determinations at concn. 100 times the detection limits. D. W. S.

1J70. **Problems in the spectroscopic analysis of acid solutions.** Arnautov, N. V.; and Kovchur, V. A. *Tr. Inst. Geol. Geofiz. Akad. Nauk SSSR, Sib. Otd.*, 1977, (315), 19–21 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G40.

1J71. **Developments in photophysical instrumentation. II. Nanosecond emission spectroscopy.** West, M. A.; and Beddard, G. S. (Davy Faraday Res. Lab., Royal Institution, London, W.1, England). *Am. Lab.*, 1976, **8** (11), 77, 78, 80, 82, 84, 86–88, 90.—The techniques of pulse-sampling, phase fluorimetry and time-correlated single-photon counting for measuring fluorescence decay profiles are compared. Free-running spark lamps and gated lamps for use as excitation sources are shown diagrammatically, as are optical and electronic systems suitable for measuring nanosecond emissions. The instrumentation necessary for obtaining time-resolved fluorescence spectra by means of the single-photon counting technique is discussed. Applications of the methods to measuring the fluorescence decay properties of small polyatomic (*e.g.*, SO_2 and biacetyl) and aromatic (*e.g.*, naphthalene) molecules in the gas phase, to the investigation of excited monomers and complexes, to the study of energy transfer and to obtain quenching curves are considered. (For Part I see *Anal. Abstr.*, 1976, **31**, 4J90.) L. B.

1J72. **'Thermostatable' micro-cells for fluorescence spectrophotometry.** Schulz, Jean-Alain [Lab. d'Acoustique Moleculaire (ERA au CNRS), Univ. Louis Pasteur, Strasbourg, France]. *J. Chim. Phys. Phys.-Chim. Biol.*, 1977, **74** (6), 721–723 (in French).—Two cells are described for measuring the frontal fluorescence of thin layers of soln.; the emission

intensity is proportional to the surface area irradiated. One cell is for equilibrium measurements on ≃0·6 ml of liquid; thermal equilibrium is attained in ≃5 s. It has been used to measure the spectra of α-chymotrypsin (**I**) and myoglobin (due to emission by tryptophan). The relationship between intensity and concn. was rectilinear for up to 4·5 mg ml^{-1} of **I**, and the intensity was twice that measured by the classical method (because of the use of a reflecting surface). The other cell is for kinetic measurements on 100 to 150 μl of liquid; thermal equilibrium is achieved within 0·5 s. It has been used to measure the thermal-relaxation kinetics of **I**.

L. B.

1J73. **Background fluorescence spectra observed in atomic-fluorescence spectrometry with a continuum source.** Fowler, W. K.; and Winefordner, J. D. (Dept. Chem., Univ. Florida, Gainesville, Fla. 32611, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 944–949.—A study is reported of such background effects in the Ar-separated air - acetylene flame under different operating conditions, with use of an EIMAC xenon-arc continuum source (pulsed and continuous). Molecular-fluorescence bands were observed and assigned to NH, CN, OH and PO (arising from the presence of PH_3 as an impurity in the acetylene and largely removed by means of a PH_3 filter). The significance of background fluorescence of this type as source of interference in a.f.s. is considered generally. R. W.

1J74. **Pulsed- *versus* continuous-wave atomic-fluorescence spectrometry.** Omenetto, N.; Boutilier, G. D.; Weeks, S. J.; Smith, B. W.; and Winefordner, J. D. (Dept. Chem., Univ. Florida, Gainesville, Fla. 32611, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1076–1078.—Information on the performance, in terms of signal-to-noise ratio, of some sources for fluorescence is collated, and some reasons for the poor performance of pulsed-fluorescence spectrometry as compared with theoretical predictions are considered. Attention is drawn to some of the drawbacks of pulsed-fluorescence methods. R. W.

1J75. **Comparison of image devices *versus* photomultiplier detectors in atomic- and molecular-luminescence spectrometry via signal-to-noise ratio calculations.** Cooney, R. P.; Boutilier, G. D.; and Winefordner, J. D. (Dept. Chem., Univ. Florida, Gainesville, Fla. 32611, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1048–1057.—A theoretical comparison is presented of three photomultiplier tubes with an image dissector, a silicon vidicon, a silicon-intensified target vidicon, an intensified silicon-intensified target vidicon and a secondary electron-conduction vidicon in respect of signal-to-noise ratio in, *e.g.*, a.f.s. Indications are given of preferred detector systems for use in different situations. R. W.

1J76. **Non-dispersive atomic fluorescence with a carbon filament as sample atomiser. I. The measuring apparatus and its use in determination of mercury.** Sanz Medel, A.; West, T. S.; and Perez-Bustamante, J. A. (Dept. Quim. Anal., Fac. Cienc. Quim., Univ. Complutense, Cuidad Univ., Madrid, Spain). *An. Quim.*, 1977, **73** (6), 849–854 (in Spanish).—The construction is described of a non-dispersive fluorescence spectrometer incorporating a carbon-filament atom reservoir source and a photomultiplier detector. Selection of the optimum experimental parameters (including radiation intensity, atomisation potential, argon flow and filament position) is discussed. Application of the technique to the estimation of Hg indicated a detection limit of 10 pg, with a coeff. of variation of 6%. Calibration graphs for up to 100 pg of Hg are presented.

J. K. C.

1J77. **Design and operation of a fluorimetric reaction-rate instrument.** Wilson, Robert L.; and Ingle, James D., jun. (Dept. Chem., Oregon State Univ., Corvallis, Ore. 97331, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1060–1065.—Details (including a block diagram and photograph) are given of the design, construction and performance characteristics of an instrument comprising a molecular-fluorescence spectrometer and a ratemeter equipped with digital read-out. Measurements of the fluorescence of quinine sulphate and of reaction rates in the 8-hydroxyquinoline-5-sulphonic acid - Ag^+ system indicate coeff. of variation of ≃0·3% for equilibrium measurements and ≃0·5% for kinetic conditions.

R. W.

1J78. **Choice between absorption and fluorescence techniques.** Hirschfeld, Tomas (Block Engineering Inc., Cambridge, Mass. 02139, U.S.A.). *Appl. Spectrosc.*, 1977, **31** (3), 245.—A quantitative comparison of the signal-to-noise ratios in the shot-noise-limited detection case indicates that fluorescence measurements will be the more sensitive, even for low fluorescence quantum efficiencies, for dilute, thin or slightly absorbing samples. When detection is limited by detector noise, absorption measurements are the more sensitive, but the techniques are equivalent for limitation by modulation noise.

M. A. P.

1J79. **Timing circuit to monitor base-line absorbance using the AS-1 graphite furnace auto-sampling system.** Epstein, Michael S. (Anal. Chem. Div., Inst. Mater. Res., Natl. Bur. Stand., Washington, D.C. 20234, U.S.A.). *At. Absorpt. Newsl.*, 1977, **16** (3), 75–76.—A simple timing circuit, based on a type NE556A dual-timer integrated circuit, is described; it enables the base-line absorbance to be read automatically at a pre-set time during the analysis cycle of a furnace atomisation system for a.a.s. The circuit was used with the Perkin-Elmer AS-1 auto-sampling system and HGA-2100 graphite furnace, and was triggered from the HGA-2100 power supply at the start of the atomisation step to give an automatic reading of the base-line absorbance 15 s later. C. K. L.

1J80. **Remote recorder timer control and recorder-pen overload-protection system for the HGA-74.** McArthur, J. H.; and Dickinson, D. R. (Dept. Earth Sci., Univ. Leeds, England). *At. Absorpt. Newsl.*, 1977, **16** (3), 73–74.—A simple timer circuit was designed for use with the Perkin-Elmer HGA-74 graphite furnace to allow control of the recorder chart drive and pen input. An electronic timing system was used to control both the delay time from programme initiation to the start of the recording period and the length of the recording period, the spans being variable between 0 and 120 s and 0 and 60 s, respectively. A switchable pen-overload-protection circuit is incorporated and consists of a relay that is actuated at all times except during the recording period and that switches in a potential divider circuit to attenuate the input signal to the recorder by a factor of 1000 in order to prevent excessive deflections of the pen. C. K. L.

1J81. **Some sources of systematic error in the determination of elements in the nanogram and picogram ranges by flameless atomic-absorption**

spectrophotometry with a graphite furnace. Volland, G.; Koelblin, G.; Tschoepel, P.; and Toelg, G. (Lab. Reinststoffe, Max Planck Inst. Metallforsch., Schwaebisch Gmuend, W. Germany). *Fresenius' Z. Anal. Chem.*, 1977, **284** (1), 1–12 (in German).—Sensitivities and limits of detection for, *e.g.*, Fe, Cd, Co, Ni can be adversely affected by the structure of the graphite, by impurities in the carrier gas, by traces of acids or of organic solvents used in the concentration of the elements to be determined, and by the heating programme itself. Low signals are specifically obtained in the presence of traces of strong acids and of halogenated hydrocarbons such as $CHCl_3$. (33 references.) H. L. L.

1J82. **Localisation of the discharge in emission-spectroscopic analysis.** Nepokoichitskii, A. G.; and Tukmachev, G. V. *Zh. Prikl. Spektrosk.*, 1977, **26** (2), 346–347 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G43.

1J83. **Plasma-torch configuration for an inductively coupled plasma as a source in optical emission spectroscopy and atomic-absorption spectroscopy.** Mermet, J. M.; and Trassy, C. (Lab. Chim. Ind. et Anal., Inst. Nat. Sci. Appl., Villeurbanne, France). *Appl. Spectrosc.*, 1977, **31** (3), 237–239.—A plasma-torch system is described that is simple to construct and has a low operating cost owing to the use of low Ar flow rates. Optimum operating parameters vary with the type of high-frequency generator used, but a general way of establishing these is outlined. M. A. P.

1J84. **Simple method and device for taking time-resolved spectra in spectrographic analysis.** Sastri, V. D. P. (Natl. Phys. Lab., New Delhi, India). *Appl. Spectrosc.*, 1977, **31** (3), 246–247.—A specially constructed Hartmann diaphragm having five vertically and horizontally displaced rectangular apertures is driven across the spectrograph slit, thereby allowing fixed exposures through different parts of the slit height during the total time of the burn. Use of this method overcomes the lateral displacement of spectra and the consequent wavelength misalignment that often occur with the racking-plate method. M. A. P.

1J85. **Effect of a controlled argon atmosphere on change in intensities of atomic and ionic lines in spectroscopic analysis of dried residues of solutions.** Krasnobaeva, N.; and Nedyalkova-Daskolova, N. *Zh. Prikl. Spektrosk.*, 1977, **26** (2), 201–206 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (14), Abstr. No. 14G78.

1J86. **Comparison of multi-channel SIT [silicon-intensified target] image vidicon and photomultiplier sequential linear-scanning systems for measurement of steady-state and transient fluorescence of molecules in solution.** Cooney, R. P.; Vo-Dinh, T.; Walden, G.; and Winefordner, J. D. (Dept. Chem., Univ. Florida, Gainesville, Fla. 32611, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 939–943.—A direct comparison is made between such systems used as detectors of fluorescence from both a simple cell and from a simulated liquid-chromatographic effluent in a flow-through cell arranged so that solutes could be injected into a steady flow of solvent. A variety of polycyclic aromatic and heterocyclic compounds, with hexane or methanol as solvent, was examined. An account is given of the fundamental characteristics involved, and it is concluded that, for steady-state fluorescence, the photomultiplier tube is slightly better, but the SIT is preferred for transient signals. With use of the SIT system, detection limits were $\simeq$20 pg for anthracene and 0·6 ng for riboflavine. R. W.

1J87. **Flame diagnostics: local temperature profiles and atomic-fluorescence intensity profiles in air - acetylene flames.** Haraguchi, H.; and Winefordner, J. D. (Dept. Chem., Univ. Florida, Gainesville, Fla. 32611, U.S.A.). *Appl. Spectrosc.*, 1977, **31** (3), 195–200.—Temperature profiles of unseparated and Ar-separated air - acetylene flames produced with a capillary burner were determined by the method previously described (Haraguchi *et al.*, *Anal. Abstr.*, 1977, **33**, 3J78). Atomic-fluorescence intensity profiles in the flames for Mg, Cu, In and Cr were also determined; these are compared with the temp. profiles, and conclusions are drawn as to the nature of the processes occurring in the flames. M. A. P.

1J88. **Measurement of an approximately 60,000:1 root-mean-square signal-to-noise ratio in Fourier-transform infra-red spectroscopy.** Foskett, C.; and Hirschfeld, Tomas (Digilab, Cambridge, Mass. 02139, U.S.A.). *Appl. Spectrosc.*, 1977, **31** (3), 239–241.—A practical demonstration is presented of the actual values of the very high signal-to-noise ratios that are attainable with use of this technique. M. A. P.

1J89. **Interpretation of infra-red and Raman spectra using pattern recognition.** Comerford, J. M.; Anderson, P. G.; Snyder, W. H.; and Kimmel, H. S. (Dept. Chem. and Chem. Eng., Inst. Technol., Newark, N.J., U.S.A.). *Spectrochim. Acta, Part A*, 1977, **33** (6–7), 651–667.—A collection of i.r. and Raman spectra of 400 organic compounds was reduced to a digital code by classifying peak intensities on a scale between 0 and 9 in 25-cm^{-1} bands from 200 to 2000 cm^{-1} and each 50-cm^{-1} band from 2000 to 4000 cm^{-1}. A computer pattern-recognition program evaluating trained vectors gave convergence with a minimum of iterative procedures when these i.r. and Raman digital codes were considered as a single concatenated vector. It correctly assigned a compound to a particular chemical class with $\simeq$90% of the 1117 spectra examined. Several examples of the use of the procedure illustrate the capability of the program to distinguish characteristic group frequencies. M. A. P.

1J90. **Micro-sampling and use of a flow cell for coherent anti-Stokes Raman spectrometry.** Rogers, L. B.; Stuart, J. D.; Goss, L. P.; Malloy, T. B., jun.; and Carreira, L. A. (Dept. Chem., Univ. Georgia, Athens, Ga. 30602, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 959–962.—A micro-cell incorporating standard m.p. capillary tubes has been applied to the measurement of coherent anti-Stokes Raman spectra excited by two tunable dye lasers. As examples, spectra for CCl_4, β-carotene, β-carboline and benzene are reproduced; tests with a flow-through system gave satisfactory results up to 900 cm s^{-1} for β-carotene. Use of the cell in a specific detector for high-resolution liquid chromatography is proposed. R. W.

1J91. **Surface and in-depth analysis by Auger-electron spectroscopy (AES).** Hofmann, S. (Max-Planck Inst. Metallforsch., Stuttgart, W. Germany). *Mikrochim. Acta*, 1977, Suppl. VII, 109–128 (in German).—A survey is given of published work,

together with examples of surface studies in the Nb - O system and of depth profiling combined with sputtering by ion bombardment. (45 references.)
J. W. P.

1J92. **Influence of the probing electron beam and sputtering in Auger-electron spectroscopy.** Braun, P.; Betz, G.; and Faerber, W. (Inst. Allg. Phys., Wien, Austria). *Mikrochim. Acta*, 1977, Suppl. VII, 129–137 (in German).—In the examination of carbonates by this technique, C cannot be detected owing to breakdown by the primary electron beam, resulting in desorption of CO and CO_2 from the surface; with silicates, low results for Na, K and Ca are caused by similar breakdown. In the single-phase alloys Ag - Au and Au - Cu there is no interference, but in the two-phase system Ag - Cu changes in peak height with sputtering are caused by diffusion of Ag.
J. W. P.

1J93. **Quantitative analysis of thin oxide films using X-ray photo-electron spectroscopy and rastered ion bombardment.** McIntyre, N. S.; and Zetaruk, D. G. (Whiteshell Nucl. Res. Establ., Atomic Energy Canada Ltd., Pinawa, Man.). *J. Vac. Sci. Technol.*, 1977, **14** (1), 181–185.

1J94. **Applications of secondary-ion mass spectrometry (SIMS).** Werner, H. W. (N. V. Philips Research Laboratories, Eindhoven, Netherlands). *Mikrochim. Acta*, 1977, Suppl. VII, 63–83.—This technique has been applied to, *e.g.*, (*i*) analysis of mono-layers for the identification of organic compounds such as PTFE; (*ii*) determination of concn. profiles of Al in surfaces of aluminium gallium arsenide and of B and F in silicon; (*iii*) imaging of surfaces to study oxide formation on stainless steel, and distribution of Fe in alumina and of Ca in biological material; and (*iv*) identification of compounds in phases in cobalt silicide multi-layers and in metals.
J. W. P.

1J95. **Test of a quantitative approach to secondary-ion mass spectrometry on glass and silicate standards.** Morgan, A. E.; and Werner, H. W. (Philips Research Laboratories, Eindhoven, Netherlands). *Anal. Chem.*, 1977, **49** (7), 927–931.—A procedure is described that permits information on composition to be derived from secondary-ion m.s. data by means of a single-parameter factor. It was applied to measurements made with a Cameca IMS 300 instrument (in some instances in an atmosphere of O) on a number of (mainly NBS) glasses and silicates; for most elements, the calculated concn. were within a factor of 2 of those obtained by other methods.
R. W.

1J96. **Acetylation as chemical-ionisation technique in medium-pressure chemical-ionisation mass spectrometry.** Hass, J. Ronald; Nixon, Willard B.; and Bursey, Maurice M. (Natl. Inst. Environ. Health Sci., Research Triangle Park, N.C. 27709, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1071–1073.—Various organic compounds were introduced, via a g.l.c. column packed with 3% of CHDMS on Gas-Chrom Q (100 to 120 mesh), into a Finnigan 1015C mass spectrometer. Acetylation spectra could be obtained by introducing biacetyl vapour (at 0·3 to 0·6 mTorr) diluted with He. Such spectra are stereo-selective and are superior in this respect to protonation spectra (*e.g.*, for *cis*- and *trans*-4-t-butylcyclohexyl acetate) and by adjustment of conditions can be made stereo-specific.
R. W.

1J97. **Collaborative study of procedure for photometric measurement in spark-source mass spectrography.** Ailloud, P.; Allegre, J.; and Richomme, C. (C.E.C.M., rue Georges-Urbain, Vitry-sur-Seine, France). *Spectra 2000*, 1977, (35), 16–24 (in French).—A photographic plate obtained in the spark-source m.s. of a steel sample was circulated to eight laboratories for calibration and measurement in a standardised manner, and the results were assessed step by step. It was concluded that, although one plate was insufficient to estimate significant differences between the participants, rigid adherence to a standard procedure offered no advantage over the use by each laboratory of its customary procedure.
R. A. C. I.

1J98. **Random and systematic errors arising during computer processing of photographed spark-source mass spectra.** Babushkina, T. V.; Glavin, G. G.; Zelenin, A. E.; and Kostenko, T. A. (Inst. Phys. Solids, Acad. Sci. USSR, Chernogolovka). *Zh. Anal. Khim.*, 1977, **32** (6), 1088–1094 (in Russian).—A procedure is described for computer processing of data obtained by densitometry of photo-plates to evaluate the sensitivity and contrast of emulsions, parameters of calibration graphs, and the results of quantitative analyses. The effects of random errors in the calculation of emulsion sensitivity and contrast and of background and maximum blackening of the photo-plate on the results of analyses are estimated. It is shown that large systematic errors in the m.s. analysis of, *e.g.*, brasses for Bi, Pb, Sb and Zn are caused by the dependence of emulsion sensitivity on the mass and energy of the various ions.
M. P.

See also Abstracts: **1A1**, Spectrophotometric methods of analysis: review. **1A2**, Mutual interference in flame photometry. **1A9**, Applications of computers in spectroscopy. **1B2**, Improved spectroscopic technique. **1B5**, X-ray fluorescence technique. **1B11**, Electron-probe analysis of films. **1B18**, Metallurgical applications of energy-dispersive micro-probe analysis. **1B23**, Study of spectrographic carriers. **1B38**, System for energy-dispersive X-ray analysis. **1B142**, Isotopic analysis of N by m.s. **1B175**, Spectroscopic methods for analysis of metals. **1B176**, Analysis of oxide films by e.s.r. **1B198** to **1B200**, Spectroscopic techniques for examination of steels. **1B215** Combination of energy-dispersive system and electron microscope for study of alloys. **1C50**, Laser system for determination of polycyclic aromatic hydrocarbons. **1C57**, Chemiluminescence detection of NO from combustion of petroleum. **1C73**, System for m.s. of dyes. **1C90**, Spectroscopic methods for determination of di-isocyanates: review. **1D14**, Atomic-emission spectrometer for determining Ba. **1F2**, Comparison of spectroscopic methods for determination of metals in turkey muscle. **1F3**, Spectroscopic methods for metals in foods: review. **1G6**, Comparison of spectroscopic instruments for analysis of soil. **1H6**, Ionisation technique for m.s. of airborne particles. **1J30**, G.c. - m.s.: review. **1J39**, Fluorescence detection in g.c. of polycyclic hydrocarbons. **1J45**, Separator for g.c. - m.s.

Radiochemistry

1J99. **Radioanalytical determinations with use of two radionuclides.** Kyrs, Miroslav; and Rais, Jiri (Inst. Nucl. Res., Rez, near Prague, Czechoslovakia).

Chem. Listy, 1977, **71** (5), 511–529 (in Czech).—A review is presented, with 100 references. J. Z.

1J100. **Improved counting phial for measurement of low-energy gamma-rays in liquid scintillation counters.** Versluijs, F. (Pharmacol. Lab., Univ., Amsterdam, Netherlands). *Anal. Biochem.*, 1977, **80** (1), 249–257.—The phial consists of two tubes, the outer one (wall thickness 1·25 mm) of borosilicate glass of low K content and the inner one (wall thickness 0·4 to 0·5 mm) of normal borosilicate glass. The inner tube is treated on the outside with silver-yellow paint and heated to 550°; at this temp., the silver diffuses into the glass to yield a deep-brown coating resistant to corrosion by scintillator soln. The space between the tubes is filled with scintillator soln., and the sample is placed in the inner tube. The phial is particularly useful for determination of the ^{125}I used in radio-immunoassays. R. A. H.

1J101. **Quench correction of single- and double-labelled samples in liquid scintillation counting by use of a versatile simple on-line computer system.** Reuter, Eckart; and Trefny, Franz (Pharmakol. Inst., Freie Univ., Berlin, W. Germany). *Int. J. Appl. Radiat. Isot.*, 1977, **28** (6), 539–544.—Instant conversion of data obtained from a liquid scintillation spectrometer to d.p.m. is achieved by use of an on-line computer system (central or desk). Samples singly or doubly labelled with ^{3}H, ^{14}C or ^{22}Na were quenched with CCl_4 or solubilised liver homogenate, and the appropriate fluor was added. Quench correction was achieved by application of the external-standard ratio method. Analysis of the counts with doubly labelled samples was carried out by the simultaneous equation method. The program permitted simple and versatile operation, and the accuracy of the results was satisfactory over a wide range of quenching. W. C. D.

1J102. **Comparison of an absolute activity analysing device with an on-line computer in liquid scintillation counting of single- and double-labelled samples.** Reuter, Eckart (Pharmakol. Inst., Freie Univ., Berlin, W. Germany). *Int. J. Appl. Radiat. Isot.*, 1977, **28** (6), 545–549.—Samples labelled with ^{3}H or ^{14}C were quenched with CCl_4, and Unisolve was added; the mixtures were then counted with a liquid scintillation spectrometer. Quench correction was applied either by (*i*) a Packard 544 absolute activity analyser (which automatically shifted the external-standard ratio to decimal values and analysed the counts for doubly labelled samples by the exclusion method) interfaced to a computer, or (*ii*) an on-line desk computer, with use of third-order polynomial coeff. and the simultaneous equation method with doubly labelled samples. For extended quench ranges, method (*i*) was preferred; however, if the quench range was divided into sections, method (*ii*) gave satisfactory results. (See previous abstract.) W. C. D.

1J103. **Calibration of lithium-drifted-germanium gamma-ray spectrometers for the measurement of radioactive noble gases.** Coursey, B. M.; Hutchinson, J. M. R.; and Unterweger, M. P. (Radioactivity Sect., Natl. Bur. Stand., Washington, D.C. 20234, U.S.A.). *Int. J. Appl. Radiat. Isot.*, 1977, **28** (6), 551–553.—A calibration graph relating full-energy-peak efficiency to γ-ray energy was prepared by use of γ-ray point source standards (mixed radionuclides) located at the 'centre' of a glass hemisphere placed at various distances above a detector (three different types were used). A glass sphere containing, *e.g.*, ^{85}Kr, was used as a gaseous source to replace the point source. An expression is presented that relates the full-energy-peak efficiency for the point source to that of the gaseous source for a given γ-ray energy. W. C. D.

See also Abstracts: **1B6**, Silica support for chromatography in radiochemical separations. **1B60**, Methods for radiochemical determination of Sr isotopes. **1F3**, Determination of trace contaminants in food by activation analysis: review.

Electrochemistry

1J104. **Electrolytic chromatography and coulopotentiography: rapid electrolysis at the column electrode used for the preparation, separation, concentration and estimation of trace and/or unstable substances.** Fujinaga, Taitiro; and Kihara, Sorin (Dept. Chem., Fac. Sci., Kyoto Univ., Japan). *Crit. Rev. Anal. Chem.*, 1977, **6** (3), 223–254.—The necessary theory and instrumentation are discussed, and applications of electrolytic chromatography at constant potential (including the determination of trace metals, of oxalate and of Pu in the presence of Ni, Cr, Cu, U, Cl and Ce) and with a potential gradient (separation of Cd, Pb and Cu; determination of ^{212}Pb, ^{212}Bi and ^{208}Tl) are reviewed. An application of coulopotentiography considered is the continuous determination of Cu, Pb and Cd, and systems for anodic-stripping coulopotentiography are discussed. (32 references.) B. H.

1J105. **Theory and applications of ion-selective electrodes. II.** Koryta, Jiri (J. Heyrovsky Inst. Phys. Chem. and Electrochem., Acad. Sci., Prague, Czechoslovakia). *Anal. Chim. Acta*, 1977, **91** (1), 1–85.—This review covers the literature from 1972 to mid-1976 and is divided into five sections as follows —theory of membrane potentials; technology of ion-selective electrodes; fixed-site ion-selective electrodes; liquid-membrane electrodes; and enzyme electrodes. The importance of plastic-film membranes is stressed, and particular attention is paid to applications in biology and medicine. (For Part I see *Anal. Abstr.*, 1973, **24**, 3265.) B. H.

1J106. **Null-point control of acidity with a differential pH electrode system.** Light, Truman S.; and Swartz, James L. (The Foxboro Co., Foxboro, Mass. 02035, U.S.A.). *Anal. Chem.*, 1977, **49** (7), 1009–1011.—Details are given of a system in which a dual high-impedance input amplifier and a glass pH-measuring electrode are used in conjunction with a reference electrode that comprises a conventional glass electrode as internal element and a filling soln. of composition similar to that of the test soln. This arrangement means that small variations in pH lead to potential changes that are less subject to errors than are those obtained with conventional pH cells. Some comparative e.m.f. values are reported for HCl, HNO_3, H_2SO_4, H_3PO_4 and acetic acid. R. W.

1J107. **Electrical conduction and analytical applications of silver-β-alumina membranes in aqueous solution.** Bernard, Claude; Bohnke, Claude; Malugani, Jean-Pierre; and Robert, Guy (Lab. Electrochim. Solides, Fac. Sci., Besancon, France). *Bull. Soc. Chim. Fr., I*, 1977, (5–6), 410–414 (in French).—These membranes were prepared by heating thin cylindrical tubes or pellets of β-$Na_2O.nAl_2O_3$ with an excess of $AgNO_3$ in an evacuated sealed tube at 310° for 24 h, then washing the product successively with H_2O, dil. HNO_3, hot conc. HNO_3 and H_2O.

The measured total and electronic conductivities show that the membranes are ionically conducting and are suitable as indicator electrodes for Ag^+ (0·1mM to 0·5M) in aq. medium. Their behaviour is similar to that of Ag_2S membranes. With measurements at pH 6 and $I = 0·1$ (adjusted with NH_4NO_3), the calibration graph was rectilinear for 0·1mM to 0·1M-Ag^+. W. J. B.

1J108. **Calibration of meter for measurement of fluoride concentration in potassium chloride background.** Komar', N. P.; and Loginova, L. P. *Zh. Fiz. Khim.*, 1977, **51** (4), 965–966 (in Russian). *Ref. Zh., Khim.*, 19GD, 1977, (15), Abstr. No. 15G160.

1J109. **Electro-deposition and stripping at graphite cloth electrodes.** Yaniv, D.; and Ariel, M. (Dept. Chem., Technion–I.I.T., Haifa, Israel). *J. Electroanal. Chem. Interfacial Electrochem.*, 1977, **79** (1), 159–167.—The suitability of graphite cloth as a porous electrode for removing or recovering trace metal contaminants from flowing aqueous media was studied using Cd, Cu and Hg. All three metals present at concn. of 10^{-4} to 10^{-5}M were deposited successfully from ammoniacal buffer soln., and the electrode maintained its porosity. Both Cd and Cu could be completely removed by anodic stripping, leaving the cloth unchanged. However, Hg was not removed even at a potential of +1 V (*vs.* the s.c.e.). Graphite cloth is considered unsuitable as an analytical electrode, but can be successfully used for the removal of dissolved contaminants. D. W. S.

1J110. **Pulsed voltammetric stripping at the thin-film mercury electrode.** Turner, John A.; Eisner, Uri; and Osteryoung, R. A. (Dept. Chem., Colorado State Univ., Fort Collins, Colo. 80523, U.S.A.). *Anal. Chim. Acta*, 1977, **90**, 25–34.—In an extension of previous work (*Anal. Abstr.*, 1977, **32**, 4B64), a mini-computer system was used to compare the merits of staircase-waveform, differential-pulse and square-wave anodic stripping for determining p.p.b. concn. of trace metals. The square-wave and differential-pulse methods (the latter with a short delay time) were the most sensitive. Because of the better sensitivity, shorter plating times can be used. W. J. B.

1J111. **Radiation-cured polymer-impregnated graphite electrodes for anodic-stripping voltammetry.** McLaren, K. G.; and Batley, G. E. (Australian Atomic Energy Comm., Res. Establ., Lucas Heights, N.S.W.). *J. Electroanal. Chem. Interfacial Electrochem.*, 1977, **79** (1), 169–178.—A study was made of impregnated graphite electrodes made from mixtures of polyester, styrene, epoxy-resins and methyl methacrylate (**I**) as alternatives to wax-impregnated graphite and vitreous-carbon electrodes in anodic-stripping voltammetry. Good electrodes, with surfaces free of voids, stable to 0·5M-acid and comparable with vitreous carbon were prepared from both dense and soft graphites impregnated with **I** or mixtures (3 : 2) of **I** with an epoxy-resin. Details of the preparation of the electrodes are given. When used for differential pulse anodic-stripping voltammetry, the limits of detection were 0·01, 0·04, 0·02 and 0·02 $\mu g\ l^{-1}$ for Cd, Cu, Pb and Zn, respectively, in 16mM-acetate buffer and 0·1mM-$Hg(NO_3)_2$ at pH 4·6. The electrodes were used for the determination of these metals in sea-water. D. W. S.

1J112. **Direct measurement of half-wave potentials.** Zheleztsov, A. V. (Moscow Energetics Inst., Kazan Branch, USSR). *Zh. Anal. Khim.*, 1977, **32** (6), 1083–1087 (in Russian).—It is shown that the $E_{\frac{1}{2}}$ values of reversible electrochemical processes can be determined by a.c. polarography with $\simeq$10-fold better precision than by conventional d.c. polarography. Expressions are given for calculating $E_{\frac{1}{2}}$ values from a.c. polarograms obtained with use of an amplitude-modulated voltage. M. P.

1J113. **Analyses in turbulent hydrodynamic voltammetric cells.** Pungor, E.; and Varadi, M. (Inst. Gen. and Anal. Chem., Tech. Univ., Budapest, Hungary). *Bull. Soc. Chim. Belg.*, 1976, **85** (11), 846–853.—The current distribution on the surface of a turbulent-flow hydrodynamic cell on which liquid, *e.g.*, the effluent from a chromatography column, impinged was measured. The cell was constructed with a silicone rubber-based graphite measuring electrode and a silver - AgCl reference electrode. Optimum responses were obtained when the electrode area was less than that of the jet of soln. The coeff. of variation of the current given by mM-$K_4Fe(CN)_6$ (8 results) was typically 1%. The system was applied to the determination of proline in aq. soln. (when O does not interfere) and to the monitoring of purine bases eluted from a dextran gel column. D. W. S.

1J114. **Processing of polarographic currents by integration.** Kalvoda, Robert (J. Heyrovsky Inst. Phys. Chem. and Electrochem., Prague, Czechoslovakia). *Chem. Listy*, 1977, **71** (5), 530–535 (in Czech).—The technique described is suitable for the amplification of low-level current and voltage signals in d.c. polarography, thereby suppressing 'noise'. The improvements achieved are illustrated by polarograms of, *e.g.*, 5μM-Tl^+ plus 15μM-Pb^{2+} in 0·1M-NaOH medium. J. Z.

1J115. **Apparatus for conductometric titration.** Levi, L. I.; Kitaev, Ya. A.; and Grigor'yan, S. A. (Steel and Alloy Inst., Moscow, USSR). *Zavod. Lab.*, 1977, **43** (6), 659–660 (in Russian).—The construction of a wide-range automatic conductivity recorder is described, with details of the electronic circuits. The apparatus provides five measuring ranges and incorporates a special circuit to maintain constant temp. in the conductivity cell. A. S. W.

See also Abstracts: **1A9**, Applications of computers in electrochemistry. **1B105**, Cell for coulometry of U and Pu. **1B107**, Electrode for titration of CN^-. **1B166**, Use of solid-electrolyte probes for determining O. **1E49**, Technique for amperometric titration of barbiturates. **1F3**, Determination of trace contaminants in food by electrometric techniques: review. **1G10**, Evaluation of electrodes for measurement of NO_3^-. **1H54**, Automatic potentiometric analyser for waste water. **1J18**, **1J19**, Electrochemical detectors for liquid chromatography.

Particle size analysis

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